INTRODUCTION

TO

CHEMICAL ANALYSIS

FOR BEGINNERS

FROM THE TENTH GERMAN EDITION

OF

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INTRODUCTION.

It is generally conceded that chemistry cannot be properly learned by the student except through the medium of individual experimental work in the laboratory. Lectures and recitations serve to present the fundamental laws of general chemistry and chemical philosophy and furnish a splendid field for experiments on an elaborate scale to illustrate these laws; but work only in the laboratory will familiarize the student with chemical changes and the properties of the elements and their compounds.

Beginners usually reason from the specific to the general, hence a clear understanding of the specific properties of a limited number of chemical elements and their compounds will lay a better foundation for an accurate and broad knowledge of the science than a more extended but less systematic course of study.

This little book originally designed for Prof. Rüdorff's pupils has met with such marked favor in Germany, having passed rapidly through 10 large editions, that the translator has been induced to offer it for the consideration of the American teacher and pupil, believing it fully fills the requirements, as herein set forth. As a guide to the student in the introductory study of the chemistry of the elements and their compounds, the popularity this work has attained at home is proof enough of its excellence.

The special feature of the book is the originality the author displays in presenting to the student chemical changes in a manner at once tangible and interesting. Pro-

fessor Rüdorff's method of teaching is best explained in his introductory remarks, as follows:

"Work in the chemical laboratory has a marked attraction for the majority of students at first; the original warm ardor, however, will often be observed to cool in consequence of the many failures incurred. Experimenting in private, without the aid of a competent teacher or guide, and students' work in the laboratory is too liable to degenerate into unsystematic testing and play with chemicals, which results only in the most discouraging failure to attain the desired object.

"The careful pursuit of elementary analytical chemistry will train the student to exact observation and correct interpretation of the same, and will give him an excellent scientific training besides developing manipulative skill. In order that this aim may not fail, the laboratory work must be based on a systematic and thorough plan. There must be a gradual succession from easy to more difficult tasks. Particular stress should be laid on this point; that the student be made to understand fully the reasons for an operation, and why it is pursued in a particular manner and not otherwise. In order to accomplish this, he must be well acquainted with the reactions of the various chemicals and have an insight into the nature of the chemical changes which take place under his observation.

"It is a well-known fact, that the beginner in analytical chemistry is often disheartened by the extent of the field to be covered, therefore, first of all, the subjects to be treated of in an elementary work should be selected within quite narrow limits.

"The author has endeavored to set forth the principles of analytical chemistry with a limited number of elements and their more important compounds. In selecting such

substances due consideration has been given to the fact of the limited facilities of most laboratories.

"The book is divided into two parts: in Part I. the reactions of the most important elements are studied, particular salts, or compounds being employed. The characteristic tests are marked with a *. The student examines the majority of the substances numbered and makes himself familiar with them (with the aid of the teacher, if necessary), and with the manipulations of reactions as well as with the nature of the chemical changes which take place. The equations are to be written out on the blank pages.

"This introductory work done, the student proceeds to Part II., which treats of the examination of given substances. This part consists of two subdivisions or chapters. The first is of a preparatory character, being confined to simple substances containing not more than one base and one acid. The metals, or bases, are NH_4, Na, K, Ba, Sr, Ca, Mg, Al, Cr, Fe, Mn, Co, Ni, Zn, Cd, Pb, Bi, Cu, Ag, Hg, Sb, As, Sn, and they may be present as oxides, sulphides, sulphates, chromates, phosphates, borates, nitrates, carbonates, arsenites, or antimonides. When the constituents are found, confirmatory tests should be made from memory or by the aid of Part I.

"After a sufficient number of analyses have been made and the remainder of Part I. has been taken up, the student, thus advanced, proceeds to the second section of Part II. which extends the limited scheme of the first section to the analysis of complex substances or mixtures of the salts containing more than one base or one acid. This plan is a little more limited, however, than the first, as regards the number of constituents allowed. Chromium and strontium are entirely excluded in complex substances, and the metals arsenic, antimony and tin, are allowed only on condition

that not more than one of them shall be present at the same time. This will also apply to the metals nickel and cobalt; neither is there allowed more than one constituent at a time which is insoluble in both water and acids. The peculiar difficulties connected with the separation and determination of the substances named will, it is hoped, be sufficient excuse for these limitations, if the aim of the book and the limited facilities, often at the command of the student, are kept in view."

In preparing the work of Prof. Rüdorff a few additions have been made which are calculated to assist the student who suffers mainly the disadvantage of being unable to devote but a small part of his time to chemical studies. As a matter of fact, the student has a great amount of trouble and sustains a loss of time from this very source, and is liable to become disheartened with chemistry in general. For his benefit, and in order to be a little more explicit, we have inserted a few short explanatory notes. The principal additions have been in giving the chemical formulas of the substances worked with and of the precipitates or new substances formed, with the exception, however, of those of indefinite compounds, such as basic salts.

THE TRANSLATOR.

LABORATORY OUTFIT.

Apparatus.

1. Test tube rack with one doz. test tubes.
2. Two small funnels.
3. Filter paper.
4. A pair of shears.
5. Wash bottle.
6. Iron ring stand, with sand bath or wire gauze.
7. Small hard-glass tubes with one end closed, two and a half or three inches long.
8. One or two small evaporating dishes.
9. Spirit lamp or Bunsen burner; adapted for a luminous flame for blowpipe experiments.
10. Blowpipe.
11. A piece of charcoal.
12. A pair of tongs or pincers.
13. Platinum foil and wire.
14. Two glass rods.

REAGENTS.

A. Liquids and Solutions.

H_2SO_4 conc.	Sulphuric Acid conc.	Spec. grav.=1.84.
H_2SO_4 dil.	Sulphuric Acid dil.	5 vol. acid : 100 water.
HCl dil.	Hydrochloric Acid.	C. P. sp. g.=1.12.
HNO_3 dil.	Nitric Acid.	C. P. sp. g.=1.20.
H_2S	Hydrogen Sulphide.	Freshly prepared saturated sol.
$(NH_4)_2S$	Ammonium Sulphide.	Yellow, 1 : 1, + ½ vol. dilute NH_4OH.
NH_4OH	Ammonium Hydrate.	1 vol. U. S. P. Aqua Am. : 1 vol. water.
NaOH	Sodium Hydrate.	Sp. g.=1.12.
$(NH_4)_2CO_3$	Ammonium Carbonate.	20 : 100 + ¼ NH_4HO
Na_2CO_3	Sodium Carbonate.	20 : 100.
Na_2HPO_4	Sodium Hydrogen Phosphate.	10 : 100.
$MgSO_4$	Magnesium Sulphate.	10 : 100 + 5 NH_4Cl.
NH_4Cl	Ammonium Chloride.	30 : 100.
$BaCl_2$	Barium Chloride.	5 : 100.

$AgNO_3$	Silver Nitrate.	5 : 100.
$K_4(FeCy_6)$	Potassium Ferrocyanide.	5 : 100.
$K_6(Fe_2Cy_{12})$	Potassium Ferricyanide.	5 : 100.
$K_2Cr_2O_7$	Potassium Dichromate.	10 : 100.
KI	Potassium Iodide.	5 : 100.
$Pb(C_2H_3O_2)_2$	Lead Acetate.	5 : 100.
$CaSO_4$	Calcium Sulphate.	Saturated Solution.
	Ammonium Molybdate.	6 : 100 + 1 HNO_3.
	Sol. Indigo.	1 : 6 H_2SO+SO_3 (Nordhausen).
$H_2C_4H_4O_6$	Tartaric Acid.	25 : 100.
$H_2C_2O_4$	Oxalic Acid.	Saturated (12 : 100).
$(NH_4)_2C_2O_4$	Ammonium Oxalate.	8 : 100.
KCyS	Potassium Sulphocyanide.	5 : 100.
$CuSO_4$	Copper Sulphate.	5 : 100.
$HgCl_2$	Mercuric Chloride.	5 : 100.
	Fuming Nitric Acid.	
	Tincture of Galls.	1 : 1 Alcohol, 4 Aq.
C_2H_5OH	Alcohol.	U. S. P.

B. Dry Reagents and Chemicals for Examination.

USED BY ALL STUDENTS IN COMMON.

1. Litmus paper, blue and red.
2. Anhydrous sodium carbonate.
3. Nitre (pulverized).
4. Borax (pulv.).
5. Copper sulphate.
6. Ferrous sulphate.
7. Manganous sulph.
8. Magnesium sulph.
9. Cadmium sulph.
10. Nickel sulph.
11. Cobalt sulph.
12. Alum.
13. Barium Chloride.
14. Ammonium chloride.
15. Sodium phosphate.
16. Lead nitrate.
17. Potassium dichromate.
18. Potassium iodide.
19. Starch.
20. Marble.

21. Strontium carbonate.
22. Minium.
23. Black oxide of manganese.
24. Arsenious acid and met. arsenic.
25. Copper turnings.
26. Tin, granulated.
27. Scrap zinc or granules.
28. Iron wire or tacks.
29. Bismuth.
30. Antimony.
31. Heavy spar (pulv.).
32. Iron pyrites (pulv.).

Certain pieces of apparatus and the majority of reagents may be employed by several students in common.

QUANTIVALENCE OF METALS.

Monovalent	Divalent.	Trivalent.	Tetravalent
K	Ba Sr	Bi	Sn (stannic)
Na	Ca Mg	Sb (antimonous)	Pt (platinic)
NH_4	Mn Zn	As (arsenious)	
Ag	Fe (ferrous)	Au	**Hexavalent.**
Hg (in mercurous compounds).	Co Ni		Double-atoms.
	Cd Pb		Fe_2
	Cu		Al_2
	Hg (mercuric)		Cr_2
	Sn (stannous)		
	Pt (platinous)		

BASICITY OF ACIDS.

Monobasic.	Dibasic.	Tribasic.	Tetrabasic.
HCl	H_2SO_4	H_3PO_4	$H_4(FeCy_6)$
HI	H_2CrO_4	H_3AsO_3	**Hexabasic.**
HNO_3	H_2CO_3	H_3AsO_4	$H_6(Fe_2Cy_{12})$

The student should remember that neglect of precautions and haste will spoil his experiments. Always keep in mind the following rules:

1. Read ahead until you understand the point and then follow out directions carefully.

2. Be on your guard, especially against employing excess of reagents or of heat. Excesses may spoil everything; insoluble substances may become soluble and *vice versa*. For instance:

Dil. HCl dissolves $BaCO_3$, but conc. HCl will not.

Dil. HCl does not dissolve PbS, but hot conc. HCl will.

$PbCl_2$ is soluble in water in presence of but little HCl, but is precipitated by addition of conc. HCl.

Most all reactions being based on the phenomena of solubility and insolubility, the importance of the above rules is apparent.

TABLE OF SOLUBILITIES.

	K	Na	NH_4	Ba	Sr	Ca	Mg	Al	Mn	Fe	Fe_2	Co
Oxide	1	1	1	1	1	1-2	2	2	2	2	2	2
Sulphide	1	1	1	1	1	1-2	2	..	2	2	..	2
Chloride	1	1	1	1	1	1	1	1	1	1	1	1
Iodide	1	1	1	1	1	1	1	1	1	1	..	1
Sulphate	1	1	1	3	3	1-3	1	1	1	1	1	1
Nitrate	1	1	1	1	1	1	1	1	1	1	1	1
Carbonate	1	1	1	2	2	2	2	..	2	2	..	2
Phosphate	1	1	1	2	2	2	2	2	2	2	2	2
Arsenite	1	1	1	2	2	2	2	..	..	2	2	2
Chromate	1	1	1	2	1-2	1-2	1	2	1	..	1	2
Borate	1	1	1	2	2	2	2	2	2	2	2	2
Oxalate	1	1	1	2	2	2	2	2	2	1-2	1-2	2

	Ni	Zn	Cd	Pb	Cu	Bi	Hg	Ag	Cr_2	Sn	As	Sb
Oxide	2	2	2	2	2	2	2	2	2 & 3	2 & 3	1-2	2
Sulphide	2	2	2	2	2	2	3	2	..	2	2	2
Chloride	1	1	1	1-3	1	2	1 & 3	3	1 & 3	1	1	2
Iodide	1	1	1	1-3	..	..	2	3	..	1	1	..
Sulphate	1	1	1	3	1	2	2	1	1	..	..	2
Nitrate	1	1	1	1	1	2	1	1	1	..	..	..
Carbonate	2	2	2	2	2	2	2	2	..	..	..	..
Phosphate	2	2	2	2	2	2	2	2	2	2	2	..
Arsenite	2	2	2	2	2	2	2	2	..	..	..	..
Chromate	2	2	2	2-3	2	2	2	2	..	..	..	2
Borate	2	2	2	2	2	2	2	2	2	2	2	..
Oxalate	2	2	2	2	2	2	2	2	1	2	2	..

The preceding table gives the solubility, in water and acids, of the most important metallic oxides, sulphides, chlorides, iodides and other salts. The meaning of abbreviations is:

1, Soluble in water.
2, Insoluble in water, soluble in hydrochloric or nitric acid.
3, Insoluble in both water and acids.
1-2, Soluble in water with difficulty, but readily in acids.
1-3, Soluble with difficulty in both water and acids.
2-3, Insoluble in water, soluble with difficulty in acids.

TO THE STUDENT.

1. Each pupil is held responsible for the condition of his desk and apparatus, and is required to leave everything clean and in good order.
2. Pupils are held responsible for breakage and the careless and wasteful use of chemicals and gas.
3. Pupils are cautioned not to mix chemicals nor to make experiments other than those given in the instructions.
4. In using reagent bottles, never lay the stoppers down; hold them between the fingers and replace at once.
5. In applying reagents, add drop by drop unless otherwise instructed.
6. Nothing should be put into reagent bottles, not even stirring rods or test paper. Never pour back a reagent once removed from its bottle.
7. Strong acids, solid matter, solutions of mercury, silver or platinum, must not be thrown into the sinks. Jars are supplied for these purposes.
8. Silver, platinum and gold slops must be saved in jars used for that purpose.
9. CAUTION. Glass and porcelain ware must be clean and dry on the outside before being heated, and must be heated carefully to avoid breakage.
10. Acids must be evaporated in the fume chambers. In evaporating solutions to dryness in glass or porcelain dishes use the water bath to avoid breakage.
11. Make your own experiments, use your own hands, eyes, reasoning faculties and common sense, and let your neighbor do the same.
12. Make your notes carefully and clear, using symbols and formulæ and equations when possible.
13. Each student should have towels and clean cloths for wiping and cleaning apparatus.
14. Follow rules carefully on page 11 in relation to reactions.

PART I.

REACTIONS.

I. COPPER.

Boil some copper turnings with hydrochloric or sulphuric acid (dil.); they remain unchanged.

Heat a few other turnings with nitric acid. The metal dissolves rapidly to blue *copper nitrate,* $Cu(NO_3)_2$, with the formation of copious reddish-brown fumes of NO_2 (the result of the reduction of HNO_3, NO being oxidized by the air into NO_2).

Copper Sulphate ($CuSO_4$).

Blue Vitriol.

Dissolve a piece of the salt the size of a bean in a test tube one-third full of water by heating, and take about one-tenth of this solution for each reaction; for reactions under headings 3-9 dilute each portion with 5-6 volumes of water before adding the reagent. The blue solution has an acid reaction, *i. e.,* turns blue litmus paper red. Reagents act upon it as follows:

REACTIONS FOR THE BASE.

1. **Zinc (or Iron)** immersed in the solution, precipitates *metallic copper* on the zinc (or iron). In the liquid, copper sulphate is replaced by zinc or iron sulphate.

*2. **Hydrogen Sulphide** solution (6-8 volumes to the copper solution), *brownish-black copper sulphide,* CuS. Heat

*Characteristic test.

the test tube gently and the precipitate will more rapidly collect and settle to the bottom upon standing. Pour off the supernatant liquid cautiously and as completely as possible, and divide the remaining liquid with the precipitate in it into two portions. To one portion add a few drops (4-5) of *nitric acid,* to the other a few drops of *hydrochloric acid,* and heat both to boiling. The precipitate remains unchanged by HCl, but dissolves in HNO_3, with the separation of a *dirty-gray sulphur* which floats on the surface of the liquid.

3. **Ammonium Sulphide** (4-5 drops) forms the same precipitate, CuS; an excess of the reagent (say 10 drops) will not dissolve the precipitate, even upon heating.

4. **Sodium or Potassium Hydrate** (10-20 drops), *blue copper hydroxide,* $Cu(OH)_2$, which on boiling is converted into *dark brown copper oxide,* CuO.

*5. **Ammonium Hydrate** (2-3 drops), *bluish-green basic sulphate (i. e.,* a combination of the neutral salt with its own hydroxide); it dissolves on the addition of more NH_4OH with *blue color.* To one drop of the original copper solution in a test tube full of water add a drop of NH_4OH; it produces a decided blue color.

6. **Ammonium Carbonate,** *bluish-green basic copper carbonate,* which is soluble in an excess of the reagent.

7. **Sodium Carbonate,** *bluish-green basic copper carbonate.*

*8. **Potassium Ferrocyanide** (a few drops), *reddish-brown* Cu_2Cfy ($=Cu_2FeCy_6$). Dilute one drop of the copper solution with a test tube full of water, and add thereto a few drops of potassium ferrocyanide. The liquid will become distinctly brown on shaking.

REACTIONS FOR THE ACID.

*9. **Barium Chloride** (a few drops) white *barium sulphate,* $BaSO_4$, which is insoluble upon addition of acids (HNO_3 or HCl).

10. Heat a small piece of blue vitriol in a glass tube closed at the lower end. It turns light gray by giving off its water of crystallization, which condenses farther up on the sides of the tube.

BLOWPIPE REACTIONS.

11. Dip the red-hot looped end of the platinum wire into powdered borax and heat what sticks to the wire in the flame of a Bunsen burner. The borax bubbles and swells up, and finally melts to a clear bead (borax bead, borax glass). Melt together with this bead a piece of blue vitriol the size of a pin head by the aid of the blowpipe. On cooling, the bead will show a *green color* from copper.

12. Mix a small crystal of blue vitriol with 4-8 parts of anhydrous sodium carbonate and melt the mixture thoroughly on a piece of charcoal before the blowpipe. A great deal of carbonic acid (CO_2) escapes during the process, the charcoal (a powerful reducing agent when hot) having combined with the oxygen of the soda and of the copper-salt, leaving the sulphur of the latter to combine with the sodium of the former producing Na_2S (Hepar). Place some of this fused mass on a *bright* silver coin and moisten with a drop of water; a *brown spot* of Ag_2S will be formed on the coin after standing. Whenever silver tarnishes, this is due to the presence of at least a trace of hydrogen or some other soluble or volatile sulphide. The copper of the blue vitriol is contained in above-named hepar as minute scales. A few larger ones may be detected with the naked eye by grinding some of the hepar in a mortar with water.

13. Heat a small piece of a copper turning before the blowpipe on charcoal. It will fuse and be burned to *black oxide,* CuO.

II. ZINC.

Pour some hydrochloric acid on a few small pieces of zinc in a test tube and *warm moderately* until the liberation of gas has ceased. The metal dissolves with the evolution of hydrogen gas and the formation of:

Zinc Chloride ($ZnCl_2$).

In order to get a solution which contains no free acid, it is necessary that there remain some undissolved zinc when the evolution of gas is over, in this solution, diluted with 6 volumes of water, reagents will produce the following changes:

REACTIONS FOR THE BASE.

1. **Hydrogen Sulphide** (6-8 vols. of the zinc solution), voluminous *white zinc sulphide,* ZnS. The precipitate dissolves readily on addition of several drops of HCl (chemical solution, resulting in the restoration of the original compound). Add to another portion of the zinc solution a few drops of HCl and then H_2S; no precipitate.

*2. **Ammonium Sulphide** (4-6 drops to the highly diluted sol.), white zinc sulphide, ZnS. To one portion of this precipitate add HCl; precipitate dissolves, but a slight turbidity remains which is due to the separation of some sulphur. Add sodium hydrate to another portion of the precipitate; it is not dissolved.

*3. **Sodium or Potassium Hydrate** (a few drops), *white zinc hydroxide,* $Zn(OH)_2$ which is redissolved on the addition of more of the reagent. From this solution H_2S throws down ZnS (comp. **XIII.**, 3).

4. **Ammonium Hydrate** (at first only a few drops, then more), the same as in 3. If previously the same vol. of ammonium chloride sol. has been added to the zinc solution, NH_4OH will not produce a precipitate. This is due to the formation of ammonium, zinc double chloride, which is unaffected by the reagent. For this reason, the NH_4OH precipitate cannot be obtained if the zinc solution contains much free acid.

5. **Sodium Carbonate,** *white basic zinc carbonate,* $ZnCO_3+2Zn(OH)_2$ (zinc carbonate, minus some of its carbonic acid, plus water=basic carbonate). Convince yourself of the liberation of CO_2 by repeating the experiment with hot zinc solution when the CO_2 will not remain absorbed in the liquid.

REACTIONS FOR THE ACID.

*6. **Silver Nitrate,** white *silver chloride,* AgCl, which on shaking collects in a curdy mass. To some of the precipitate add a few drops of ammonium hydrate, to the balance a few drops of nitric acid; it will rapidly dissolve in the former, but not in the latter.

BLOWPIPE REACTIONS.

7. Fuse a small piece of zinc on charcoal before the blowpipe. The zinc burns with the formation of white fumes, which are partly deposited on the coal. This "coating" ZnO is yellow while hot and white when cold.

8. *Fuse some zinc salt with about 6 parts of dry sodium carbonate on charcoal before the blowpipe; zinc is reduced from the compound (comp. I., 13), but no faster than it burns off, forming a coating of ZnO beyond the flame. For this reason no globule of metal can be found in the fused mass.

III. MANGANESE SULPHATE ($MnSO_4$).

Dissolve as much of the salt as the point of a knife will hold in a test tube full of water by the aid of heat. The solution has an acid reaction. From the solution, diluted with water, there will be precipitated by:

REACTIONS FOR THE BASE.

1. **Hydrogen Sulphide** nothing.

*2. **Ammonium Sulphide** (a few drops) flesh *colored manganese sulphide,* MnS, which turns dark brown by oxydation upon a short exposure to the air.

*3. **Sodium or Potassium Hydrate,** white *manganese hydrate,* $Mn(OH)_2$, which, in contact with the air, turns brown.

4. **Ammonium Hydrate** produces the same precipitate.

5. **Sodium Carbonate,** white *manganese carbonate,* $MnCO_3$.

REACTIONS FOR THE ACID.

*6. **Barium Chloride,** white *barium sulphate,* $BaSO_4$, insoluble in HCl, a few drops (comp. I., 9).

BLOWPIPE REACTIONS.

7. If a little of the salt is fused with 6 parts of dry sodium carbonate on charcoal before the blowpipe, the fused mass will blacken a moist silver coin (comp. I., 12).

8. *Ignite an exceedingly small piece of the salt on platinum foil over a Bunsen burner, with a mixture of 2 parts of sod. carb. and 1 part of nitre. It gives a *dark green fusion* of *potassium manganate,* K_2MnO_4. If too much of the manganese compound has been used the color of the fusion will be so intense as to appear black.

9. *The borax bead will be colored *violet* by a very small piece of the salt.

IV. IRON.

Warm several small tacks or pieces of iron wire with HCl in a test tube until the evolution of gas has completely ceased; pour off the solution from the undissolved iron and divide it into 2 parts. The black floating mass is undissolved carbon. The solution contains:

Ferrous Chloride ($FeCl_2$).

Dilute one part of the solution with 10 volumes of water. In this solution reagents will produce the following changes:

REACTIONS FOR THE BASE.

1. **Hydrogen Sulphide** no change.

*2. **Ammonium Sulphide** (3-5 drops), *black ferrous sulphide,* FeS. Add several drops of HCl to some of the precipitate; it dissolves; but a slight, permanent turbidity remains, due to the separation of sulphur.

3. **Sodium or Potassium Hydrate,** *greenish ferrous hydrate,* $Fe(HO)_2$, which, in the upper part of the tube, where it comes into contact with the air, will become a dirty green and finally brown, owing to the formation of ferric hydrate.

4. **Sodium Carbonate** precipitates *white ferrous carbonate,* $FeCO_3$, which, in contact with the air, turns dirty green and finally brown (formation of ferric hydrate).

5. **Potassium Ferrocyanide** (a few drops), a *light blue precipitate* which turns darker by contact with the air.

*6. **Potassium Ferricyanide,** a *dark blue precipitate* of $Fe_3(Cfy)_2(=Fe(FeCy_6)_2)$ Turnbull's blue.

To the other part of the ferrous chloride solution add 8-10 drops of nitric acid and 2-3 drops of hydrochloric acid, and boil. The former serves to oxydize the H of the latter

to H_2O and set free Cl. The *ferrous* is converted into yellowish red *ferric chloride.* The intermediate black color of the liquid is due to the absorption of nitrogen oxide, NO (reduced from HNO_3, see I.) in an, as yet, unoxidized portion of the ferrous solution. Boil, if necessary, with addition of a few drops of HNO_3, until a drop of the fluid diluted with some water will yield a reddish-brown precipitate with sodium hydrate. Be careful not to use more HNO_3 than is absolutely necessary. If this should have been the case, however, neutralize the excess of acid by adding NH_4OH drop by drop; but not so much as to produce a precipitate that will not redissolve upon shaking. Reagents will produce the following reactions in this solution of

Ferric Chloride (Fe_2Cl_6).

REACTIONS FOR THE BASE.

*7. **Hydrogen Sulphide,** slightly yellow or white sulphur with the reduction of the ferric or ferrous solution.

8. **Ammonium Sulphide** (4-6 drops) *reduces* the *ferric* to *ferrous* solution, and then precipitates *black ferrous sulphide,* FeS. If the solution is extremely dilute, NH_4HS will color it only a dark green. If there is much free acid in the solution, the FeS precipitate will disappear on shaking and more NH_4HS must be added.

*9. **Sodium or Potassium Hydrate,** *yellowish-brown ferric hydrate,* $Fe_2(OH)_6$, which is insoluble in an excess of the reagent.

10. **Sodium or Ammonium Carbonate,** *yellowish-brown,* $Fe_2(OH)_6$; CO_2 escapes.

*11. **Potassium Ferrocyanide** (a few drops) precipitates *Prussian blue,* $Fe_4Cfy_3[=Fe_4(FeCy_6)_3]$. Even very dilute solutions will be colored a distinct blue by this re-

agent. Liquids to be tested with Ky_4Cfy must be but slightly acid, else errors will occur.

12. **Potassium Ferricyanide** colors the solution dark only or brown without producing a precipitate (comp. ferrous reaction).

*13. **Potassium Sulphocyanide** colors the solution *blood-red (extremely delicate test).* The solution must contain some free acid.

14. **Tannic Acid or Tincture Galls** throws down *dark ferric tannate* (ink is often made from this).

BLOWPIPE REACTIONS.

15. Before the blowpipe, iron burns to *ferro ferric* or *magnetic oxide,* Fe_3O_4 (blacksmith's scale). This or a small piece of copperas (ferrous sulphate) colors the borax bead in the reducing flame *greenish,* in the oxydizing flame *yellow* to brown, which, on cooling, gradually becomes lighter colored.

V. LEAD.

Lead is scarcely affected by hydrochloric or sulphuric acids, but dissolves in warm nitric acid with the liberation of red fumes (comp. I.) and the formation of:

Lead Nitrate ($Pb(NO_3)_2$).

Dissolve as much of the crystallized salt as the point of a knife blade will hold in a test tube full of water by the aid of heat. This solution, diluted with 1 vol. of water, will give the following reactions:

REACTIONS FOR THE BASE.

*1. **Hydrogen Sulphide** (6-8 vols.), *black lead sulphide,* PbS. The same reagent, however, may also produce a red precipitate: Add a few drops of HCl to some of the lead

solution, and, if a white precipitate ($PbCl_2$) should form, dissolve it by the aid of heat. From this solution a few drops of H_2S will precipitate *red* $PbCl_2+3PbS$, which, on further addition of H_2S, will be converted into black PbS. Warm the liquid with the PbS precipitate, allow the latter to subside, and decant the supernatant liquid. To one part of the precipitate add a few (4-5) drops of nitric acid, to another part a few drops of hydrochloric acid, and boil. The precipitate dissolves in HNO_3 with the separation of sulphur, but remains unchanged in HCl.

2. **Ammonium Sulphide** produces the same precipitate. PbS.

3. **Sodium or Potassium Hydrate,** *white hydroxide,* $Pb(OH)_2$, which dissolves on warming with more of the alkali.

4. **Sodium Carbonate,** *white basic lead carbonate.*

*5. **Sulphuric Acid** dil., white *lead sulphate,* $PbSO_4$, which, on the addition of several drops of HCl or HNO_3, remains undissolved. If the solution contains ammonium salts or very much free acid the $PbSO_4$ will separate out only after the addition of alcohol (no chemical change, only physical insolubility).

*6. **Hydrochloric Acid or a Chloride,** white lead chloride, $PbCl_2$. Add NH_4OH to one-half of the precipitate, it remains unchanged (comp. **IX.**, 5, **XVI.**, 4). Heat the other half with half a test tube full of water. The $PbCl_2$ will dissolve, but on cooling crystallize out again in shining needles.

7. **Potassium Iodide** (6-10 drops) throws down yellow *lead iodide,* PbI_2. Fill the test tube with water, heat to boiling and filter off quickly from the remaining PbI_2. The dissolved PbI_2 crystallizes out on cooling in glistening gold-yellow scales.

8. **Potassium Dichromate** (a few drops), yellow *lead chromate,* $PbCrO_4$ (chrome yellow), which dissolves on the addition of sodium hydrate (especially upon warming), but is not dissolved by dilute HNO_3.

9. Fill a test tube with a diluted solution of the lead salt and suspend in it a strip of a zinc sheet. Metallic lead will be precipitated on the zinc strip in the form of glistening scales or leaves (lead tree).

REACTIONS FOR THE ACID.

*10. **A Copper turning** and several drops of conc. sulphuric acid, on being heated with some of the lead solution, evolve red fumes, which are recognized by their smell and color as NO_2. (The precipitate produced by the H_2SO_4 does not interfere with the reaction.) (Comp. I.)

*11. Add a few drops of indigo solution and some HCl to 8-10 drops of the lead solution. The mixture will be decolorized on heating.

BLOWPIPE REACTION.

12. Fuse some of the lead salt with sodium carb. on charcoal before the blowpipe. A yellow deposit is formed beyond the fusion, and the latter contains soft, malleable globules of lead. Lead melts very easily before the blowpipe, and deposits a yellow coating of PbO (litharge).

VI. RED LEAD, MINIUM (Pb_3O_4).

Warm a little of the red powder with *water.* It remains undissolved and unchanged.

*1. Take the point of a penknife full of minium and heat with HCl (10-12 drops). Cl gas will be evolved and the minium converted into white $PbCl_2$, which is soluble in a large quantity of warm water.

2. Dilute 1 vol. *nitric acid* with 4 vol. water and pour

this dilute acid on a penknife-point full of minium in a test tube. It will be partly dissolved, especially on heating. A residue of brown dioxide, PbO_2, is left, which will quickly settle to the bottom. The supernatant liquid contains *nitrate of lead* and gives all the reaction of lead (comp. **V.**).

3. Heat a little minium in a glass tube; it will be converted into litharge (pale yellow when cold) and give off oxygen gas.

4. Minium, before the blowpipe on charcoal, is reduced to metallic lead forming in globules.

VII. BISMUTH.

Heat a knife-point full of the pulverized metal with a little conc. HNO_3. Continue heating with the occasional addition of small portions of conc. HNO_3, until *almost* all of the metal has dissolved. This solution of

Bismuth Nitrate ($Bi(NO_3)_3$)

which should not be diluted with water, gives the following reactions:

REACTIONS FOR THE BASE.

*1. Water (8-10 vols.) precipitates white *basic bismuth nitrate,* $Bi(NO_3)(OH)_2$. If previously several drops of conc. sol. of *sodium* or *ammonium chloride* have been added to the bismuth solution, water precipitates bismuth *oxychloride* (pearl white), Bi OCl, which is insoluble on addition of tartaric acid (comp. **XI.**, 1). If the bismuth solution contains too much free acid, even a very large quantity of water will not produce a precipitate. (The amount of water necessary to sufficiently decrease the activity of the liquid for a precipitation of basic salt is, at the same time, sufficient to dissolve most or all of the latter, in spite of its

being only slightly soluble. For this reason any excess of acid in preparing the bismuth solution should be avoided.)

*2. **Hydrogen Sulphide,** dark brown *bismuth sulphide,* Bi_2S_3.

3. **Ammonium Sulphide** the same; insoluble in excess of the reagent.

4. **Sodium, Potassium or Ammonium Hydrate,** *white hydroxide,* $Bi(OH)_3$, insoluble in excess of the reagent (comp. **V.**, 3).

5. **Ammonium Carbonate,** *white basic carbonate,* Bi_2CO_5 ($=(BiO)_2CO_3$).

6. **Potassium Dichromate,** *yellow chromate,* $Bi_2Cr_2O_9$, ($=(BiO)_2Cr_2O_7$), soluble in dil. HNO_3, but insoluble in NaHO or KHO (comp. **V.**, 8).

BLOWPIPE REACTION.

7. Bismuth melts before the blowpipe and gives a dark yellow coating and a *brittle* metallic globule (comp., **V.-VI.**).

VIII. BARIUM CHLORIDE ($BaCl_2$).

Dissolve a knife-point full of the salt in a test tube full of water, by the aid of heat. This solution gives the following reactions:

REACTIONS FOR THE BASE.

1. **Hydrogen or Ammonium Sulphides or Ammonium Hydrate** nothing.

*2. **Ammonium or Sodium Carbonate,** *white barium carbonate,* $BaCO_3$.

*3. **Sulphuric Acid** dil., or a sulphate, precipitates, even from a very dilute solution, *white barium sulphate,* $BaSO_4$, insoluble on addition of HNO_3 or HCl.

*4. **Calcium Sulphate** sol. (5-6 vols.) gives immediately *a white turbidity* of $BaSO_4$.

5. **Sodium Hydrogen Phosphate** precipitates $BaHPO_4$, soluble in HCl.

Ammonium Oxalate, *white barium oxalate,* BaC_2O_4.

7. **Potassium Chromate** (or dichromate, made alkaline by NH_4HO), *yellow barium chromate,* $BaCrO_4$, soluble on addition of few drops of HNO_3 or HCl (comp. **V.**, 8, **VII.**, 6).

REACTION FOR THE ACID.

*8. **Silver Nitrate,** *white, curdy* AgCl; the precipitate collects on shaking; it is insoluble in HNO_3 (several drops), but dissolves readily in NH_4HO (10 to 15 drops).

9. Heat some barium chloride in a glass tube; it gives off its water of crystallization.

BLOWPIPE REACTION.

10. It melts on charcoal before the blowpipe; the fused mass, placed on red litmus paper and moistened with one drop of water, gives an alkaline reaction.

11. *Dip the hot loop of a platinum wire into the pulverized salt, and then introduce it into the flame of a Bunsen burner or a spirit lamp. The flame will be colored *yellowish-green.*

IX. MERCURY (QUICKSILVER).

Dilute a little nitric acid (conc.) with the same volume of water and gently warm a few drops of mercury with this dil. acid; the metal will dissolve to *mercurous salt* with the liberation of brown fumes of NO_2. To obtain a solution perfectly free from mercuric salt it is necessary that about half of the mercury remain undissolved, after the action of the acid is over (when large gas bubbles are colorless). Dilute this turbid solution of:

Mercurous Nitrate ($Hg_2(NO_3)_2$)

with 5-6 vol. of water, allow to settle and use the clear liquid for the following reactions:

REACTIONS FOR THE BASE.

*1. **Copper** precipitate *metallic mercury,* which is deposited on the copper and amalgamates with it. Dip a copper cent or a small, bright piece of copper sheet or wire into the diluted solution (1 drop sol., 10 drops water), taking care, however, that only part of the copper be immersed. It becomes white, amalgamating as far as it dips under the liquid. Brighten the amalgamated part by gently rubbing with a small piece of filter paper. The mercury will volatize and the copper reassume its red color if the amalgamated metal is heated in a flame and the red color of the copper reappears.

*2. **Hydrogen or Ammonium Sulphide,** *black mercurous sulphide,* Hg_2S; the precipitate is insoluble in warm dil. HNO_3, as well as in ammonium sulphide.

*3. **Sodium or Potassium Hydrate,** *black mercurous oxide,* Hg_2O.

4. **Ammonium Hydrate** throws down a *black amido compound.* (In the presence of HCl or chlorides $(NH_2Hg_2)Cl$).

*5. **Hydrochloric Acid or a Chloride,** *white mercurous chloride,* HgCl (Calomel) which is turned black by addition of NH_4OH (comp. **VI.**, 6, and **XXI.**, 4).

6. **Potassium Iodide** (a few drops), *greenish-yellow mercurous iodide,* HgI.

7. **Potassium Dichromate,** *red mercurous chromate,* Hg_2CrO_4.

8. Dissolve the residue of mercury (left from above) to *mercuric nitrate* by warming with HNO_3 conc. This solu-

tion still contains more or less mercur*ous* nitrate. In order to complete the oxydation, boil with the occasional addition of small quantities of HNO_3 conc., until a drop of the solution, diluted with water, does not react, *i. e.*, become turbid on the addition of a drop of HCl. This solution of

Mercuric Nitrate ($Hg(NO_3)_2$)

diluted with 5-6 vols. of water, gives the following reactions:

REACTIONS FOR THE BASE.

*8. **Copper,** *metallic mercury;* same as for mercurous solution.

*9. **Hydrogen (or Ammonium) Sulphide** (drop by drop, shaking between successive additions) first white $Hg(NO_3)_2+2HgS$; as more H_2S is added the precipitate turns yellow, orange, brownish, and is finally converted into black, pure mercuric sulphide, HgS, by an excess of the reagent. Mercuric sulphide does not dissolve in dilute warm HNO_3.

*10. **Sodium or Potassium Hydrate** precipitates *yellow mercuric oxide,* HgO.

11. **Ammonium Hydrate** (a few drops), a *white amido compound* $(NH_2Hg)NO_3$. In the presence of large quantities of ammonic salts (from too much free acid in the mercuric nitrate sol.) the precipitate either does not form or dissolves in the excess of NH_4OH.

12. **Sodium Carbonate,** brownish-yellow basic carbonate.

13. **Hydrochloric Acid,** no change.

14. **Potassium Iodide** (add by drops) *scarlet-red mercuric iodide,* HgI_2 soluble in excess of reagent.

15. **Potassium Dichromate,** yellowish-red mercuric chromate. ($HgCr_2O_7$.)

16. Mix a little of a dry mercury salt with dry sodium

carb. and a little charcoal powder and heat the mixture in a glass tube. A mirror, composed of minute drops of mercury, will be deposited on the sides of the tube. The metallic nature of the deposit becomes apparent on rubbing the globules together with a splinter of wood or a glass rod.

X. TIN.

Warm some granulated tin or a few tin turnings for a long time with conc. HCl or until the liberation of hydrogen gas has ceased and the greater part of the metal has been dissolved. Heat gently, to avoid the boiling away of the HCl. The solution contains:

Stannous Chloride ($SnCl_2$)

and gives the following reactions:

REACTIONS FOR THE BASE.

*1. **Hydrogen Sulphide,**—*dark-brown stannous sulphide,* SnS. Warm the fluid with the precipitate in it for a little time, and allow it to stand several minutes or until the precipitate has settled to the bottom. Pour off the clear liquid as completely as possible and *warm* (not boil) the precipitate with 10-12 drops of yellow ammonium sulphide. The stannous sulphide will dissolve to ammonium sulphostannate after being converted into *first* stannic sulphide. The latter may be obtained as a yellow precipitate on the addition of HCl to the well diluted sulphostannate solution.

*2. **Ammonium Sulphide** (a few drops) produces the same precipitate as above, which is soluble in an excess.

3. **Sodium Hydrate,** *white stannous hydrate,* $Sn(OH)_2$, soluble in excess of the alkali.

4. **Sodium Carbonate,** *stannous hydrate,* $Sn(OH)_2$, with escape of CO_2.

5. **Mercurium Chloride [or Nitrate]** (2-3 drops) either white *calomel* (Hg_2Cl_2), or gray *metallic mercury,* depending on the quantity of the reagent and stannic chloride, $SnCl_4$.

Boil the remainder of the tin with HNO_3. The metal will be converted into white metastannic acid, $H_{10}\ Sn_5O_{15}$, without being dissolved.

Dissolve completely a little gran. tin in the least possible quantity of aqua regia (1 vol. HNO_3+3 vols. HCl) by heating. Use fresh quantities of acid only when the liquid gets turbid or the metal no longer dissolves. This solution contains *stannic* and *stannous* chloride. Complete the oxydization by adding a few drops of HNO_3 conc. and heating until a drop of the liquid, diluted with water, gives a *light yellow* precipitate with H_2S. This solution which contains exclusively:

Stannic Chloride ($SnCl_4$),

diluted with 5-6 vols. of water, gives the following reactions:

REACTIONS FOR THE BASE.

*6. **Hydrogen Sulphide** (8-10 vols.), *yellow stannic sulphide,* SnS_2, soluble in ammonium sulphide. Add HCl to this well diluted solution and the precipitate will reappear (comp. **X.**, 1). To a portion of the stannic solution first add about 3 vols. of a saturated solution of oxalic acid and then H_2S, *no precipitate* is formed. If less oxalic acid is used, H_2S colors the solution yellow and a precipitate will form after a time, but more rapidly on warming.

*7. **Ammonium Sulphide** (several drops to the very dilute solution) produces the same precipitate.

8. **Sodium Hydrate,** *white stannic hydrate,* H_2SnO_3, soluble in excess of alkali to *sodium stannate,* Na_2SnO_3.

BLOWPIPE REACTION.

9. Tin is easily fusible before the blowpipe and burns to white SnO_2.

XI. ANTIMONY.

Heat a small portion of the pulverized metal with HCl; it remains unchanged. Boil another small portion with HNO_3; the metal is converted into white oxide, but not dissolved.

Warm a penknife-point full of pulverized antimony with several drops of aqua regia (1 vol. HNO_3, 3 vols. HCl) and add as much of the mixture as is required to dissolve *almost* all of the metal. This solution of:

Antimony Trichloride ($SbCl_3$),

which should not be diluted with water,—gives the following reactions:

REACTIONS FOR THE BASE.

*1. **Water** (8-10 vols.), *white oxychloride*, SbOCl, Tartaric acid, if previously added, prevents the precipitation by water (comp. **VII.**, 1).

*2. **Hydrogen Sulphide** precipitates *orange-yellow* antimony trisulphide, Sb_2S_3, which dissolves in yellow ammonic sulphide (comp. **X.**, 1) to sulphantimonite and sulphantimonate. Hydrochloric acid precipitates from this well diluted solution a mixture of Sb_2S_3 and Sb_2S_5 (golden sulphuret). Oxalic acid does not prevent precipitation by H_2S. (Comp. **X.**, 6.)

3. **Ammonium Sulphide** produces the same precipitate as H_2S.

4. **Sodium Hydrate,** *white antimonous hydrate,* $HSbO_2$, soluble in excess of reagent.

5. **Ammonium Hydrate** gives $HSbO_2$, insoluble in ex-

cess. Previous addition of tartaric acid prevents precipitation.

BLOWPIPE REACTIONS.

6. Antimony melts in a glass tube without volatilizing.

*7. It melts on charcoal before the blowpipe and evolves copious heavy fumes of antimonous oxide, Sb_2O_3, which deposits a *white coating* on the charcoal and which crystallizes around the bead on cooling. Fuse a piece of Sb, the size of a match-head before the blowpipe on charcoal and while very hot quickly throw it out on the table, it spatters and the small globules formed, in running out, leave white tracks, from Sb_2O_3, in their wake.

XII. ARSENIOUS ACID OR ANHYDRIDE (As_2O_3).

Warm a penknife-point full of the white arsenic in a test tube full of water for a time (10 min.). A solution is obtained which will scarcely redden blue litmus paper. It gives the following reactions:

REACTIONS FOR THE BASE.

*1. **Hydrogen Sulphide** (8-10 vols.) turns the solution yellow only. From this liquid, however, the addition of a few drops of HCl will throw down *yellow arsenic trisulphide,* As_2S_3, which dissolves readily in ammonium sulphide or carbonate. Yellow As_2S_3 is re-precipitated from these (well diluted) solutions on supersaturating them with HCl. Previous addition of oxalic acid *does not* prevent the precipitation of arsenic by H_2S (comp. **X.**, 6).

*2. **Ammonium Sulphide** (2-4 drops) will not precipitate anything; an addition of HCl is necessary to precipitate As_2S_3.

3. **Silver Nitrate** (2-3 drops), no reaction. Neutralize the arsenious acid with 1 drop of NH_4OH and a *yellow* pre-

cipitate of *silver arsenic,* Ag_3AsO_3, is produced. It is readily soluble in both free NH_4OH and HNO_3.

4. **Copper Sulphate** (2-3 drops), no precipitate. Addition of 1 drop of NH_4OH will throw down *green copper arsenite,* $CuHAsO_3$ (Scheele's green) Paris green, which dissolves with *blue color* in excess of the NH_4OH.

5. Metallic arsenic dissolves on being boiled with aqua regia. Heat some As, in a closed glass tube; it volatilizes without melting previously, and deposits a mirror of the metal on the sides of the tube.

BLOWPIPE REACTIONS.

*6. Heat a very small quantity of As before the blowpipe on charcoal. It burns to *white* As_2O_3, accompanied by a garlic-like odor (due to fumes of metallic arsenic).

*7. Place some As_2O_3 (the size of a pin-head) into the bottom of a closed glass tube, and some powdered charcoal on top of it. Heat very slowly from top downward, and a mirror of As will be deposited above the charcoal.

8. As_2O_3 on charcoal before the blowpipe gives white fumes and garlic-like odor.

XIII. POTASSIUM ALUMINIUM SULPHATE ($K_2Al_2(SO_4)_4$).

(Alum.)

Dissolve a large knife-point full of the salt in a test tube of water by the aid of heat. It dissolves readily in hot, but with great difficulty in cold water. The solution reddens blue litmus paper and gives the following reactions:

REACTIONS FOR THE BASE.

1. **Hydrogen Sulphide** nothing.

*2. **Ammonium Sulphide** (3-4 drops), white, pasty gelatinous *aluminium hydrate,* $Al_2(OH)_6$, with the liberation of H_2S.

*3. **Sodium Hydrate** (a few drops), the same precipitate. Add more alkali and the precipitate will dissolve. Divide this solution of *sodium aluminate,* $Na_6Al_4O_9$, into 2 parts, and add to one ammonium chloride, to the other hydrogen sulphide. Ammonium chloride decomposes the aluminate and reprecipitates *aluminium hydrate,* whilst hydrogen sulphide has no effect. (Comp. **II.**, 2.)

*4. **Ammonium Hydrate** gives *aluminium hydrate,* insoluble in excess of reagent. If tartaric acid is added to a solution of alum, neither sodium, potassium nor ammonium hydrate will produce a precipitate.

5. **Sodium Carbonate** precipitates *aluminium hydrate* with the liberation of carbonic acid gas.

6. **Sodium Hydrogen Phosphate** throws down white *aluminium phosphate* $Al_2(PO_4)_2$, which dissolves in excess of sodic hydrate, but is insoluble in ammonium hydrate, except in presence of tartaric acid (comp. 3 and 4).

REACTION FOR THE ACID.

7. **Barium Chloride** gives barium sulphate (comp. **I.**, 9).

BLOWPIPE REACTION.

8. Alum upon being heated in a glass tube gives off water.

9. Fuse a little alum with 6 parts of sod. carb. before the blowpipe on charcoal. The fusion blackens moist silver. (Comp. **I.**, 12.)

10. *Introduce a small piece of the salt on platinum wire into a Bunsen or spirit lamp flame. The flame becomes *violet* (potassium flame).

XIV. CALCIUM CARBONATE ($CaCO_3$).

Marble, Limestone, Chalk.

Calcium carbonate is insoluble in water, but dissolves in hydrochloric acid, with the liberation of a colorless and odorless gas; carbonic acid gas, CO_2. In order to obtain a neutral solution, warm a piece of marble the size of a bean with hydrochloric acid until the evolution of gas has ceased. Finally heat to boiling in order to expel the carbonic acid gas absorbed by the liquid. Take, however, only as much acid as will leave some of the marble undissolved. A burning splinter of wood will be extinguished if plunged into the upper part of the test tube during the evolution of CO_2.

When all of the carbonic acid gas is expelled, dilute with water; this is a neutral solution of

Calcium Chloride ($CaCl_2$).

It gives the following reaction:

REACTIONS FOR THE BASE.

1. **Hydrogen or Ammonium Sulphide** nothing.

2. **Ammonium Hydrate** nothing.

*3. **Ammonium or Sodium Carbonate,** *white calcium carbonate,* $CaCO_3$.

*4. **Sulphuric Acid,** dil. *white calcium sulphate,* $CaSO_4$ (Gypsum). No precipitate will form, however, if the calcium solution is largely diluted with water previous to adding sulphuric acid. If half vol. of alcohol is added to this solution of calcium sulphate, the precipitate will separate out at once upon shaking (comp. **VIII.**, 3 and 4).

*5. **Ammonium Oxalate** throws down *calcium oxalate,* CaC_2O_4. The precipitate dissolves on addition of hydrochloric acid.

6. **Sodium Hydrogen Phosphate,** *white calcium phosphate,* $CaHPO_4$.

7. **Potassium Dichromate,** no precipitate in both neutral and ammoniacal calcic solutions (comp. **VIII.**, 7, and **XV.**, 5).

BLOWPIPE REACTION.

8. Ignite a small piece of marble, the size of a mustard seed, before the blowpipe on charcoal. CO_2 escapes and the residue has an *alkaline reaction, i. e.,* turns blue a piece of moist red litmus paper.

XV. STRONTIUM CARBONATE ($SrCO_3$).

Insoluble in water, but soluble in hydrochloric acid with the liberation of carbonic acid gas, CO_2 (comp. **XIV.**).

STRONTIUM CHLORIDE ($SrCl_2$).

This solution gives the following reactions:

REACTIONS FOR THE BASE.

1. **Hydrogen or Ammonium Sulphide or Ammonium Hydrate** nothing.

*2. **Sulphuric Acid,** dil. *white strontium sulphate,* $SrSO_4$. In very dil. solutions the precipitate appears only after shaking (comp. **VIII.**, 3 and 4).

*3. **Calcium Sulphate** renders the solution turbid only after some shaking.

4. **Sodium Hydrogen Phosphate** white *strontium phosphate,* $SrHPO_4$, soluble in hydrochloric acid.

5. **Potassium Dichromate** no precipitate in dilute ammoniacal solution (comp. **VIII.**, 7, and **XIV.**, 7).

*6. Dip a platinum wire in the strontium solution and introduce into the flame, it is *colored a beautiful red.*

XVI. AMMONIUM CHLORIDE (NH_4Cl.).

(Sal-ammoniac.)

The solution has a neutral reaction.

REACTIONS FOR THE BASE.

1. **Hydrogen or Ammonium Sulphide, Ammonium or Sodium Carbonate,** produce no precipitate.

2. **Tartaric Acid** throws down from concentrate solutions white crystalline *Ammonium hydrogen tartrate,* $NH_4H(H_4C_4O_6)$. Shaking facilitates the formation of the precipitate, especially in case of a somewhat dilute solution. Precipitate soluble in hydrochloric acid and in sodium hydrate.

*3. Heat a small knife-point full of the salt with sodium hydrate. The gas discharged at the mouth of the test tube smells of *ammonia,* NH_3, and on coming into contact with moist red litmus paper, *colors it blue.*

REACTION FOR THE ACID.

*4. **Silver Nitrate** precipitates silver chloride, AgCl (dilute solution previously). Precipitate soluble in ammonium hydrate; insoluble in nitric acid.

XVII. POTASSIUM NITRATE (KNO_3).

(Nitre, Saltpeter.)

Dissolve a knife-point full of the salt in a test tube half full of water. The solution has a neutral reaction.

REACTION FOR THE BASE.

1. **Tartaric Acid** (conc.) precipitates from it potassium hydrogen tartrate, ($KHC_4H_4O_6$, cream of tartar). Precipitate dissolves in acids (hydrochloric) and in sodium hydrate with the formation of Rochelle salt, $KNaH_4C_4O_6$.

REACTIONS FOR THE ACID.

*2. A *strip of copper,* brought into the solution heated with conc. sulphuric acid, liberates reddish-brown fumes (comp. **V.**, 10).

*3. **Solution of Indigo** is decolorized by heating with the solution which has been previously mixed with hydrochloric acid (comp. **V.**, 11).

4. Heat a small piece of nitre before the blowpipe on charcoal. The result is a violent deflagration. The residue contains potassium carbonate and has an alkaline reaction, *i. e.*, colors *red* litmus paper *blue.*

5. **Nitre fuses** on being heated in a glass tube. Oxygen is evolved.

*6. Nitre colors a flame *violet (Potassium flame).*

XVIII. MANGANESE DIOXIDE, (MnO_2).

1. The mineral ground to a fine powder is insoluble in water and dil. sulphuric or nitric acid.

2. Boil a small portion of it with hydrochloric acid. It dissolves to *manganese chloride,* $MnCl_2$, with the liberation of chlorine. The solution, after filtering from the undissolved powder, may be used for repeating the reactions on manganese (comp. **III.**, 1-5).

3. Manganese dioxide is infusible before the blowpipe on charcoal.

4. Add an exceedingly small quantity of the mineral to a borax bead. It will be colored violet.

*5. Fuse a minute quantity of the mineral with sod. carb. and nitre on platinum foil. A green fusion is obtained (comp. **III.**). If too much of the manganese compound is used, the color is so intensely dark green that it appears black.

XIX. MAGNESIUM SULPHATE ($MgSO_4$).

(Epsom Salts.)

Dissolve a large knife-point full of the salt in a test tube full of water with the aid of heat. The solution has an acid reaction.

REACTION FOR THE BASE.

1. **Hydrogen (or Ammonium) Sulphide** precipitates nothing.

2. **Sodium Hydrate** throws down *magnesium hydrate,* $Mg(OH)_2$.

*3. **Ammonium Hydrate** the same. If, however, ammonium chloride, or some other ammonium salt, has been added previously to the solution, *ammonium hydrate will not throw down anything* (comp. II., 4), hence, previous addition of any acid will likewise prevent a precipitation.

4. **Sodium Carbonate** throws down a *basic carbonate* which is soluble in ammonium salts.

*5. **Ammonic Carbonate** nothing (comp. 3).

*6. **Sodium Hydrogen Phosphate** throws down a *crystalline precipitate* of *ammonium magnesium phosphate,* $MgAmPO_4$, *from solutions containing ammonium chloride (or other ammonium salts) and ammonium hydrate.* In very dilute solutions the precipitate forms only on shaking.

REACTIONS FOR THE ACID.

*7. **Barium Chloride,** barium sulphate, $BaSO_4$, insoluble in HCl.

8. **Lead Acetate,** *lead sulphate,* $PbSO_4$, insoluble in dil. acids (nitric).

On heating the salt in a glass tube water is given off.

XX. SODIUM HYDROGEN PHOSPHATE (Na_2HPO_4).

Dissolve a knife-point full of the salt in a test tube full of water.

The solution has an alkaline reaction and nothing is precipitated by tartaric acid, alkaline sulphides or carbonates. The solution gives the following reactions with:

REACTIONS FOR THE ACID.

*1. **Silver Nitrate,** *yellow silver phosphate,* Ag_3PO_4. Precipitate readily soluble in both $(NH_4)OH$ and nitric acid.

2. **Lead Acetate,** white *lead phosphate,* $Pb_3(PO_4)_2$, soluble in nitric acid, insoluble in $(NH_4)OH$.

3. To 1 drop of solution added to ¼ test tube of water, a few drops of HNO_3 and then 10-12 drops of *ammonium molybdate.* The solution will turn *yellow upon heating,* and a yellow precipitate will form after standing.

4. **Barium Chloride,** *barium phosphate,* $BaHPO_4$.

*5. **Magnesium Sulphate,** on *previous addition of ammonium chloride and hydrate,* throws down $MgNH_4PO_4$. If solution is very dilute the precipitate separates out only after shaking.

On heating the salt in a glass tube it gives off water.

*A colorless *flame becomes yellow* if sodium phosphate is introduced *(sodium flame).*

XXI. SILVER.

Dissolve a small silver coin in the least possible amount of *nitric acid* with the aid of heat. The blue solution contains the *nitrates of silver and copper.* Dilute it with a large amount of water and precipitate the silver as silver chloride, by adding hydrochloric acid drop by drop, as long as a reaction takes place. The turbid liquid will clear up on shaking; the white, curdy precipitate settles rapidly; the supernatant clear liquid solution contains *copper nitrate,* and when decanted from the AgCl, may be used to repeat the

reactions for copper (comp. **I.**). Fill the test tube or containing vessel with water, shake well and decant from the AgCl. Wash the precipitate two or three times more in the same manner, all the copper solution will thus be removed from the precipitate. Dissolve the washed AgCl by adding a *small* quantity of NH_4OH and immerse a little zinc in the solution. The AgCl will be reduced to *metallic* Ag after 24 hours. Remove the undissolved zinc and wash the metallic silver by repeatedly heating it with water (the first previously acidulated with a little sulphuric acid); finally, dissolve the pure silver by heating it with the least possible amount of HNO_3. The solution contains:

Silver Nitrate ($AgNO_3$).

The diluted solution gives the following reactions:

REACTIONS FOR THE BASE.

1. **Copper** precipitates *crystalline metallic silver.* The solution turns blue from copper nitrate, $Cu(NO_3)_2$. Dilute a few drops of the silver solution and place a little piece of copper in it. Let it stand 24 hours.

*2. **Hydrogen or Ammonium Sulphide,** *black silver sulphide,* Ag_2S.

3. **Sodium Hydrogen Phosphate** (a few drops), yellow *silver phosphate,* Ag_3PO_4, which dissolves readily in NH_4OH (a few drops) and in HNO_3; therefore, no precipitate is formed if the above solution contains free nitric acid.

*4. **Hydrochloric Acid or a Chloride,** *white, curdy* AgCl, which is blackened on exposure to the light. It dissolves readily in NH_4OH, but not in HNO_3 (a few drops) (comp. **V.**, 6, and **IX.**, 5).

5. **Potassium Iodide** (a few drops) *light-yellow silver iodide,* AgI, insoluble on addition of HNO_3 or NH_4OH.

XXII. POTASSIUM DICHROMATE ($K_2Cr_2O_7$).

Dissolve a crystal of the salt the size of a bean in ½ test tube full of water; divide the solution into 2 parts and dilute one of them with still more water. From this dilute solution there is precipitated by:

1. **Hydrogen Sulphide,** *grayish-green chromium hydrate,* $Cr_2(OH)_6$, mixed with sulphur. The solution contains (yellow) potassium chromate, K_2CrO_4. Ammonium sulphide acts in the same way. If HCl is added to the original solution previous to the addition of H_2S, sulphur only will be thrown down. All the chromate is reduced to chromium salt, and the liquid turns green, especially upon heating.

REACTIONS FOR THE ACID.

2. **Lead Acetate,** *yellow lead chromate,* $PbCrO_4$, soluble in KHO, insoluble in dil. HNO_3.

3. **Potassium, Sodium or Ammonium Hydrate or Sodium Carbonate** change the red color of the solution into yellow (formation of K_2CrO_4 from $K_2Cr_2O_7$).

4. **Potassium Dichromate** is readily reduced to chromium salt, *e. g.*, by H_2S in acid solution (comp. **I.**), or on addition of sulphurous acid sol. (H_2SO_3) (to *potassium chromium sulphate,* chromium alum). Heat the second part of original solution with several drops of hydrochloric acid. Add alcohol drop by drop and keep the liquid warm. A fruity odor is given off; the chromate is reduced and the emerald green liquid contains:

Chromium Chloride (Cr_2Cl_6).

REACTIONS FOR THE BASE.

5. **Hydrogen Sulphide** precipitates nothing.

*6. **Ammonium Sulphides** throw down *grayish-green chromium hydrate,* $Cr_2(OH)_6$.

*7. **Sodium Hydrate,** *chromium hydrate,* which dissolves with green color on the addtion of more of the alkali. Boiling will reprecipitate the chromium hydrate.

8. **Ammonium Hydrate** produces the same precipitate ($Cr_2(OH)_6$), which after long standing (in excess of NH_4OH), will dissolve more or less with a reddish violet color.

9. **Sodium Carbonate** precipitates a *greenish-gray, basic carbonate.*

BLOWPIPE REACTION.

*10. Fuse a small particle of potassium dichromate or some other chromium compound in a borax bead. It will be *colored emerald green.*

*11. Filter off precipitate obtained with sodium carbonate and fuse some of it on platinum foil with sod. carb. and nitre. The fusion is *colored yellow* with K_2CrO_4.

*12. Boil some conc. solution of a chromate with conc. hydrochloric acid. Chlorine is evolved and Cr_2Cl_6 is formed.

XXIII. SODIUM TETRABORATE ($Na_2B_4O_7$).

(Borax.)

Dissolve a piece of borax the size of a bean in a test tube one-half full of water. The solution has an alkaline reaction.

REACTION FOR THE ACID.

1. **Conc. Hydrochloric or Sulphuric Acid** throws down boric acid, H_3BO_3; as the liquid cools the precipitate will become more copious.

2. **Barium Chloride,** *barium borate,* BaB_2O_4, soluble in large volume water, especially upon heating.

Borax gives off water on being heated in a glass tube.

*3. Introduce borax into the flame by means of a looped platinum wire; it bubbles and swells up and finally melts to

a clear bead *(borax bead).* The flame shows the yellow *sodium color* during this process.

*4. Moisten a borax bead with a drop of conc. sulphuric acid and introduce it into the *edge* of the flame. At first the flame is colored *green from boric acid,* later on the yellow sodium color prevails (comp. **VIII.**, 11).

XXIV. POTASSIUM IODIDE (KI).

Dissolve a piece of the salt the size of a bean in a test tube full of water. From this solution there is precipitated by:

REACTIONS FOR THE ACID.

*1. **Lead Acetate,** *yellow lead iodide,* PbI_2, soluble in a large amount of hot water. Crystallizes out on cooling (comp. **V.**, 7).

2. **Mercuric Chloride or Nitrate,** *scarlet red, mercuric iodide,* HgI_2, (comp. **IX.**, 14).

*3. **Silver Nitrate,** *yellow silver iodide,* AgI, insoluble in HNO_3 and NH_4OH.

*4. **Starch Paste** and a drop of dil. fuming nitric acid color the very dilute solution a *deep blue.* This blue color disappears on heating, but reappears on cooling. Instead of fuming nitric acid 1 drop of HCl and 1 drop potassium permanganate, $K_2Mn_2O_8$, may be used.

5. Potassium iodide, on being fused with the addition of a minute piece of potassium dichromate, gives off *violet vapors* of Iodine. The same effect is obtained by heating potassium iodide with a little manganese dioxide and H_2SO_4 conc.

6. The Bunsen flame is *colored violet* by the salt *(potassium flame).*

XXV. CADMIUM SULPHATE ($CdSO_4$).

Dissolve a piece of the salt the size of a bean in a test tube full of water by the aid of heat. This solution gives the following reactions:

REACTIONS FOR THE BASE.

1. **Zinc,** *metallic cadmium* (comp. **V.**, lead-tree).

*2. **Hydrogen Sulphide,** *yellow cadmium sulphide,* CdS.

3. **Ammonium Sulphide** the same precipitate, insoluble in excess of ammonium sulphide (comp. **X.**, 6, and **XII.**, 1).

*4. **Sodium Hydrate,** *white cadmium hydrate,* Cd $(OH)_2$, insoluble in excess (comp. **I.**, 4, and **XIX.**, 3).

*5. **Ammonium Hydrate** the same; insoluble in excess of reagent. Ammonium hydrate (or carbonate), however, will form no precipitate in cadmium solutions if a large quantity of an ammonium salt has previously been added (comp. **II.**, 4). Sodium hydrate precipitates cadmium hydrate from the ammoniacal cadmium solution, even in the presence of ammonium salts. Warming facilitates this reaction.

6. **Ammonium Carbonate,** *white cadmium carbonate,* $CdCO_3$, insoluble in excess of reagent.

REACTION FOR THE ACID.

7. **Barium Chloride,** *barium sulphate,* $BaSO_4$.

8. Fuse some of the salt with dry sol. carb. on charcoal before the blowpipe. A rainbow-colored coating of CdO is deposited on the charcoal without the formation of metallic globules (comp. **II.** and **V.**).

XXVI. NICKEL SULPHATE ($NiSO_4$).

The green solution of the salt reacts as follows:

REACTIONS FOR THE BASE.

1. **Hydrium Sulphide,** no reaction.

*2. **Ammonium Sulphide,** *black nickel sulphide,* NiS,

which is insoluble in dil. hydrochloric and in acetic acids.

3. **Sodium Hydrate,** *pale green hydroxide,* $Ni(OH)_2$.

4. **Ammonium Hydrate** the same precipitate, soluble in excess of NH_4OH with pale blue color.

*5. **Sodium Carbonate,** *apple-green basic carbonate.*

6. **Ammonium Carbonate** the same; the precipitate is soluble in excess of ammonium carbonate, with a *bluish-green color.*

REACTIONS FOR THE ACID.

Barium Chloride, *white,* $BaSO_4$.

7. The salt loses water on being heated in a glass tube.

8. The fusion obtained with sod. carb. before the blow-pipe on charcoal blackens moist silver.

9. The borax bead is *colored reddish-brown* by a little fragment of the salt.

XXVII. COBALT SULPHATE ($CoSO_4$).

The reddish solution reacts as follows with:

1. **Hydrogen Sulphide,** no reaction.

*2. **Ammonium Sulphide,** *black cobalt sulphide,* CoS, insoluble in dil. HCl.

3. **Ammonium Hydrate,** a *blue basic salt;* after some time it dissolves in excess of NH_4OH with a brown color.

4. **Sodium Carbonate,** *a basic carbonate.*

*5. **Ammonium Carbonate,** a basic salt of *peach blossom color;* it is soluble in an excess with a violet-red color.

6. **Sodium Hydrogen Phosphate,** *violet-blue phosphate,* $Co_3(PO_4)_2$.

*7. The salt, even in minute quantities, *colors the borax bead blue.*

XXVIII. IRON PYRITES (FeS_2).

1. Warm a small knife-point full of the pulverized mineral with HCl; it *remains unchanged.* Boil a similar

portion with HNO_3; it *dissolves* with the liberation of reddish-brown fumes and the separation of gray flakes of sulphur.

This solution, diluted with water, shows all the reactions for ferric compounds (**IV.**, 7-14) *and* H_2SO_4.

2. *Heat strongly* a little of the powder in a glass tube; sulphur sublimes and the residue is *soluble in HCl* with the liberation of H_2S. The latter is recognized by its smell and its action (blackening) on filter paper moistened with lead acetate solution.

3. *Ignite* a small piece of pyrite before the blowpipe on charcoal. It burns with a pale blue flame which smells of SO_2. The residue fuses and is then *soluble in HCl* with the evolution of H_2S. If the test is *fused for a long* time it is *converted into magnetic oxide of iron,* Fe_3O_4.

4. A sample of the pulverized mineral with dry sod. carb. before the blowpipe on charcoal gives a fusion which blackens moist silver.

5. Heat iron filings and sulphur together in a test tube a compound of iron sulphide is formed. This dissolves easily in dil. H_2SO_4 or HCl, with evolution of H_2S. The solution gives all the reactions for ferrous compounds (comp. **IV.**, 1-7).

XXIX. BARIUM SULPHATE ($BaSO_4$).

(Heavy Spar.)

The finely pulverized mineral is insoluble in water and in all acids.

1. In order to produce a soluble compound from the insoluble $BaSO_4$ boil a knife-point full for some time (10-15 minutes) with a saturated solution of Na_2CO_3 (20-30 c. c.). Na_2SO_4 and $BaCO_3$ are produced. Filter off the residue, wash it well, reject the wash water, and pour dilute warm

HCl on the filter. *The $BaCO_3$ dissolves in the acid and the filtrate contains $BaCl_2$ and gives the reactions of* **VIII.**

2. The liquid obtained by boiling the powder with Na_2CO_3 contains, besides the latter, Na_2SO_4. *Acidulated with HCl, it gives the reactions for sulphuric acid* (comp. I., 9).

3. Fuse some of the barium sulphate with dry sod. carb. on charcoal. The fusion contains Na_2S, which blackens moist silver (comp. I.).

PART II.

SYSTEMATIC COURSE OF QUALITATIVE ANALYSIS.

In examining any substance for its various constituents certain reagents must be used systematically. By so doing only is it possible to state with certainty that the substance under investigation contains certain constituents and that others are not present. It is not by any means sufficient to show that this or that element is present, but it is just as *necessary to prove that these alone and no others are present.*

The work of examining a given substance is divided into two parts, viz.: examination for *bases or electropositive* and *acids or electronegative* constituents. The determination of the bases is to precede the examination for acids. In examining for the latter, *first consider which of the acids may be present, taking into account the constituents and properties of the substances already determined.*

PAY SPECIAL ATTENTION TO THE FOLLOWING POINTS:

1. In substances soluble in water one cannot expect to find an acid, which forms a compound, insoluble in water, with one of the bases determined: thus, *e. g.,* the presence of a heavy metal or metal of the alkaline earths in a substance soluble in water will exclude the presence of carbonic, phosphoric or boric acid; or, barium or lead being present in a substance soluble in water or hydrochloric acid, there is no necessity to test for sulphuric acid.

2. Be careful that the reagent intended to detect the

acid may not give a precipitate with one of the metals present; *e. g.*, in testing a silver-bearing substance for sulphuric acid one ought not to use barium chloride as a reagent, because its chlorine would give a precipitate just as well with the silver as the barium would with the sulphuric acid. In this case barium nitrate should be used instead of barium chloride or the silver should be removed by hydrochloric acid before adding the barium chloride.

3. Be careful not to test a substance for constituents which have been introduced as reagents during previous tests, *e. g.*, do not test a substance dissolved in hydrochloric acid for chlorine. Substances to be tested for chlorides should not be dissolved in hydrochloric but in nitric acid.

4. In testing a solution, first add but a few drops of the reagent, and a little more only if a precipitate is obtained.

5. Never add ammonium sulphide or carbonate to a solution containing free acid, but first ammonium hydrate to perfect neutrality or until liquid smells of ammonia after shaking, no matter whether this addition causes a precipitate or not.

6. In testing a solution with hydrogen sulphide sol. use at least 5 vols. of the latter. Ammonium sulphide, however, should be added, drop by drop, only, to the liquid previously neutralized with ammonium hydrate.

In order to examine any substance, heat no more than a small knife-point full with about one-third test tube full of water; if this does not dissolve the substance at all, or only partially, heat another portion equally as large with 10-15 drops of hydrochloric acid and then add a little water. If the above fails to produce a solution, try a third portion with nitric acid. *But always avoid excess of acids.* If even now the substance does not dissolve it has to be regarded as in-

soluble in water and acids. Accordingly it will be practical to divide all substances into three classes, viz.: Those

SOLUBLE IN WATER,
SOLUBLE IN ACIDS ONLY,
INSOLUBLE IN WATER AND IN ACIDS.

Sometimes a substance will not be dissolved by either water or acids, but will be changed; a fact which should be carefully noted, as it may give a valuable hint for the detection of certain constituents.

DETECTION OF THE BASES.

The bases with regard to their behavior towards the most important reagents may be divided into 5 groups. The CHIEF REAGENTS are the following four:

1. **Hydrogen Sulphide.** 2. **Ammonium Sulphide.** 3. **Ammonium Carbonate.** 4. **Sodium Hydrogen Phosphate.**

The following table shows, at a glance, the classification into 5 groups. From the solution, acidulated with a few drops of hydrochloric acid, there is precipitated by:

Hydrogen Sulphide		*Ammonium Sulphide	Ammonium Carbonate	Sodium Hydrogen Phosphate	Not Precipitated.
Precipitate is, in $(NH_4)_2S$,					
Soluble	Insoluble				
Sb	Hg	Fe	Ba	Mg	K
As	Pb	Co	Sr		Na
Sn	Ag	Ni	Ca		NH_4
	Cu	Mn			
	Bi	Cr			
	Cd	Zn			
		Al			

These reagents, as well as those given later on, are to be used in invariably the same order, and the following reagent

*Add first NH_4OH to alkaline reaction.

is to be used only if the preceding one either gave no precipitate, or if all the precipitable matter has been thrown down by it and filtered off.

Before starting on the examination in the wet way, it is very useful to make a few blowpipe tests. These so-called preliminary tests often give important hints, in many cases prove the presence of certain substances with perfect distinctness, or, at least, make their presence probable.

PRELIMINARY TESTS.

1. Heat a sample of the substance (the size of a pinhead) in a glass tube, and see whether there is any sublimate to be distinguished or water given off.

2. Ignite in a glass tube a like quantity with dry sodium carbonate and charcoal powder. (If the substance contains water, remove it by previously heating moderately and cautiously on platinum foil.) The formation of a metallic ring is an indication of arsenic or mercury (see **IX.** and **XII.***).

3. Heat a small quantity of substance on charcoal before the blowpipe. There may be a deflagration, a characteristic smell or a coating (comp. **XVII.**, **XII.**, **V.**).

4. Heat a small quantity of substance with dry sod. carb. before the blowpipe on charcoal and observe: coating, metallic globules and fused mass (comp. **I.**, **III.**, **V.**, **VI.**, **VII.**, **XXV.**).

After having detected by the aid of the following scheme the constituents of a substance, it is very useful to make several confirmatory tests, such as are given in the "REACTIONS." For this reason in the subsequent chapter, when an element is indicated, reference is made to the corresponding number in the "REACTIONS."

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*Subjects under Part I., "Reactions."

CHAPTER I.

SECTION 1.

EXAMINATION OF SIMPLE SUBSTANCES.

(CONTAINING AT MOST 1 BASE AND 1 ACID.)

COMPOUNDS SOLUBLE IN WATER.

A. Bases.

OUTLINES OF THE PROCESS.

Dissolve a small knife-point full of the substance to be examined in a test tube one-third full of water, with the aid of heat.

Add some 6 or 8 drops of hydrochloric acid to about one-quarter of above solution (a precipitate thus formed would indicate a lead, silver or mercurous salt) and then, no matter whether hydrochloric acid has produced a precipitate or not, add:

Hydrogen Sulphide.

If a precipitate is formed examine it according to (page 78) ..... I.

If hydrogen sulphide produced no precipitate add to a second portion of original solution some ammonium chloride and ammonium hydrate until the fluid smells of it after shaking; and, no matter whether ammonium hydrate produced a precipitate or not, add a few drops of

Ammonium Sulphide.

If a precipitate is formed, examine it according to (page 82) ..... II.

If ammonium sulphide also produces no precipitate,

add to a third portion of original solution ammonium chloride and ammonium hydrate and

Ammonium Carbonate.

If a precipitate is produced examine it accordinig to (page 82) ..... **III.**

If, however, no precipitate forms in the above liquid add to it several drops of

Sodium Hydrogen Phosphate.

If a precipitate forms, see page 84..... **IV.**

If even after shaking no precipitate appears, examine the fourth portion of the original solution according to (page 84) ..... **V.**

I. The Precipitate Obtained by Hydrogen Sulphide may be:

1. **White.** The precipitate is either separated sulphur and due, probably, to the presence of a ferric salt (comp. **IV.**, 7) or it may be zinc sulphide, due to too small an amount of free acid. Reject this precipitate and carry out exact examination for iron and zinc in the ammonium sulphide precipitate (see **II.**, page 82).

2. **Yellow or Yellowish Red** and will be CADMIUM, STANNIC, ARSENOUS OR ANTIMONOUS SULPHIDE. Heat liquid with precipitate and allow it to stand until settled. Then pour off supernatant liquid as completely as possible and warm (not boil) precipitate for a few minutes with 10-15 drops of yellow ammonium sulphide:

(**a.**) *Precipitate does not dissolve.* The original substance on being heated with sod. carb. before blowpipe on charcoal will give a rain-bow colored coating (comp. **XXV.**) ..... **Cadmium.**

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*"Reactions," Part I.

(**b.**) *Precipitate dissolves:* presence of tin, antimony and arsenic.

(1a.) Original solution gets turbid on addition of a large volume of water. Substance on being heated with sod. carb. before blowpipe on charcoal gives a white coating (comp. **IX.**) ..... **Antimony.**

(2a.) Precipitate some of original solution with hydrogen sulphide, and remove supernatant liquid. The precipitate is soluble in ammonium carbonate on warming. Original substance, heated before blowpipe on charcoal gives off a garlic-like smell (comp. **XII.**) ..... **Arsenic.**

(3a.) Hydrogen Sulphide will not produce a precipitate in original solution on previous addition of 3 vols. of conc. oxalic acid solution (comp. **X.**, *stannic salt*) .... **Tin.**

3. Black or Brown, and will be STANNOUS, LEAD, COPPER, BISMUTH, MERCURY OR SILVER SULPHIDE.

a. *Precipitate dissolves* on being heated with yellow ammonium sulphide, and hydrochloric acid throws down from this solution a yellow precipitate (comp. **X.**, *stannous salt*) ..... **Tin.**

b. Original solution with a large volume of water gives a white turbidity (comp. **VII.**) ..... **Bismuth.**

c. Original solution gives a white precipitate with dil. sulphuric acid (comp. **V.**) ..... **Lead.**

d. Hydrochloric acid causes a white precipitate in original solution which:

(1a.) Dissolves in ammonium hydrate (**XXI.**) . . **Silver**

(2a.) Is blackened by ammonium hydrate (**IX.**, *mercurous salt*) ..... **Mercury.**

e. Original solution (blue or green) is colored blue by ammonium hydrate (**I.**) ..... **Copper.**

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*Refer back to page 78 I.

f. Original solution yields a yellow precipitate on addition of sodium hydrate; a bright strip of copper turns white in the dil. orig. solution (**IX.**, *mercuric salt*) . . . . **Mercury.**

II. The Precipitate Obtained by Ammonium Sulphide may be:

1. **Black**, and will be IRON, NICKEL OR COBALT SULPHIDE.

a. *Precipitate dissolves readily in dil. hydrochloric acid;* the diluted solution yields a dark blue precipitate with potassium ferricyanide (**IV.**, 6) . . . . . **Iron.**

b. *Precipitate did not dissolve;* but sodium carbonate gives with the original solution:

(1a.) A rose-red precipitate and orig. substance colors borax bead blue (**XXVII.**) . . . . . **Cobalt.**

(2a.) A light green precipitate (**XXVI.**) . . . . . **Nickel.**

2. **Light Colored**, and will be MANGANESE, OR ZINC SULPHIDE OR CHROMIUM OR ALUMINIUM HYDRATE.

a. Orig. substance fused with sod. carb. and nitre on platinum foil gives a green fusion (**III.**) . . . . . **Manganese.**

b. Orig. substance fused with sod. carb. and nitre on platinum foil gives a yellow fusion (**XXII.**) . . . . **Chromium.**

c. Orig. solution gives with sodium hydrate a white or grayish-white precipitate, dissolved by addition of an excess of sodium hydrate. From this solution will be thrown down by:

(1a.) †Hydrogen sulphide a white precipitate (**II.**) . . . . . **Zinc.**

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†Note.—If the solution effected by sodium hydrate is not perfectly clear, filter before adding hydrogen sulphide or ammonium chloride; also warm after having added the same.

*Refer to page 78 I.

(2a.) Nothing by hydrogen sulphide; but a white precipitate by ammonium chloride (**XIII.**)..... **Aluminium.**

III. The Precipitate Obtained by Ammonic Carbonate may contain Barium, Strontium or Calcium. The original solution will give:

1. **A white precipitate** with CALCIUM SULPHATE SOLUTION which:

a. *Appears at once.* Potassium dichromate, neutralized with ammonium hydrate, produces a yellow precipitate in orig. solution (**VIII.**)..... **Barium.**

b. *Does not form until after some time.* Original solution gives no precipitate with potassium dichromate and ammonium hydrate. Original substance colors a flame red (**XV.**)..... **Strontium.**

2. **No Reaction** with CALCIUM SULPHATE.

Original solution gives a white precipitate with ammonium oxalate (**XIV.**)..... **Calcium.**

IV. If the Preceding "Chief Reagents" have not Affected the Original Solution, add some Ammonium Chloride, excess of Ammonium Hydrate and some Sodium Hydrogen Phosphate.

If a precipitate forms (perhaps *not without* shaking), it shows the presence of (**XIX.**)..... **Magnesium.**

V. Original Solution, having given no Reaction with preceding Reagents:

1. On being boiled with potassium hydrate, smells of ammonia (**XVI.**) ..... **Ammonium.**

2. Original substance on being introduced by platinum wire into the edge of a Bunsen flame, colors the same

a. Yellow (**XX.**) ..... **Sodium.**

b. Violet (**XVII.**) ..... **Potassium.**

B. ACIDS.

1. Pour some hydrochloric or nitric acid upon some of the original substance. If the fluid effervesces and the escaping gas is:

a. *Colorless and odorless,* it is carbonic acid (**XIV.**). Substance is a..... **Carbonate.**

b. *Smelling like rotten eggs* and blackening paper soaked with lead acetate sol. hydrogen sulphide (**XVIII.**), the substance is a..... **Sulphide.**

2. Acidulate the well diluted orig. solution with hydrochloric or nitric acid and add barium chloride. If *a white precipitate* is formed (**I.**, 9) the substance is a... **Sulphate.**

3. Moisten the loop of a platinum wire with conc. sulphuric acid, then dip into the substance under examination and introduce into the edge of the Bunsen flame (**XXIII.**), if the latter becomes green, the substance is a..... **Borate.**

4. Add ammonium chloride, an excess of ammonium hydrate and some magnesium sulphate to orig. solution. A white precipitate (often only after shaking) proves the substance to be a (**XX.**, 5)..... **Phosphate.**

5. †Original solution, acidulated with nitric acid, gives with argentic nitrate:

a. A white curdy precipitate, readily soluble in ammonium hydrate (**VIII.**, 8). Substance is a..... **Chloride.**

b. A yellowish precipitate, which does not dissolve on

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†Note.—If solution contains a metal precipitated by ammonium hydrate, it must be removed by some appropriate process before adding silver nitrate.

addition of ammonium hydrate. Original solution becomes blue on addition of some starch paste and a few drops of dil. fuming nitric acid (**XXIV.**). Substance is an..... **Iodide.**

6. If above tests do not determine the acid, pour some conc. sulphuric acid on a little of the original substance (no matter whether this produces a precipitate or not), add a copper turning, and heat.

Or, add a little hydrochloric to a few drops of original solution and a few drops of sol. indigo, and heat. If red fumes of well known smell appear, in the first, and if the liquid is decolorized in the second case, the substance is a ..... **Nitrate.**

SECTION No. 2.

SIMPLE SUBSTANCES, SOLUBLE IN ACIDS ON

A. Bases.

OUTLINE OF THE PROCESS.

If the substance is insoluble in water heat a sma point full of it for a few minutes with some 10 or 1 of hydrochloric acid. When dissolved, heat to boil add water. If any of the substance remains und pour off the above solution and repeat the operatio fresh portion of hydrochloric acid. When everythin solved, heat to boiling before diluting the solution. I chloric acid does not dissolve the substance try nitric

The solution obtained is to be examined in t manner as given above for *"Substances Soluble in* Attention needs only be called to a precipitate fo diluting the original solution with water (indicatio muth or antimony). *The above scheme (page 82), l*

requires to be modified if the ammonium sulphide precipitate is light colored, as it then need not necessarily contain one of the metals under **II.**, 2, but may contain one of the metals under **III.** and **IV.**, *provided the same be present as a phosphate or borate.* The phosphates or borates are re-precipitated as light-colored substances from their solution in free acid as soon as the latter is neutralized by the addition of ammonium hydrate and ammonium sulphide.

Phosphoric acid can be determined by adding to the solution 1 or 2 drops of HNO_3 and an excess of ammonium molybdate and gently warming, a yellow color on precipitate indicates phosphoric acid (comp. **XX.**, 3). Boric acid may be determined by testing the original material on platinum wire with H_2SO_4 in the flame.

In the case of simple substances insoluble in water, soluble in acids, portion **II.** *of the scheme* (SECTION 1) *will read as follows:*

II. The Precipitate obtained by Ammonium Sulphide may be:

1. **BLACK**, and will be SULPHIDE OF IRON, COBALT OR NICKEL; is to be examined as above (page 82).

2. **Light Colored or White**; PHOSPHORIC OR BORIC ACID MAY BE PRESENT. The precipitate will contain one of the metals MANGANESE, CHROMIUM, ALUMINIUM, ZINC, MAGNESIUM, CALCIUM, STRONTIUM, BARIUM. Add dilute sulphuric acid and alcohol to original solution and shake.

A. *No precipitate is formed;* no calcium, strontium or barium present, but one of the following metals: Mn, Cr, Al, Zn, Mg.

a. Test with sod. carb. and nitre on platinum foil for (see page 82) ..... **Manganese.**

b. Test with sod. carb. and nitre on platinum foil for (see page 82).....**Chromium.**

c. Original solution gives a precipitate with sodium hydrate which, in excess of reagent, is:

1a. Soluble; see page 82; test for..**Aluminium and Zinc.**

2a. Insoluble (a phosphate or borate of)...**Magnesium.**

B. *A precipitate is formed;* presence of either barium, strontium or calcium (as a phosphate or borate). In the original diluted solution dil. sulphuric acid will produce a precipitate

a. At once .....**Barium.**

b. Only after some shaking. The original substance moistened with hydrochloric acid imparts to a Bunsen flame red the color of a salt of.....**Strontium.**

c. Not at all. The addition of alcohol to this liquid will produce a white precipitate.....**Calcium.**

If ammonium sulphide produces no precipitate, barium, strontium, calcium, magnesium, cannot be present as a phosphate or borate. Consequently, the examination is continued according to **III.** and **IV.**, (Sec. 1.) as in page 82-84.

V. need not be considered, as all alkaline salts are soluble in water.

B. Acids.

In testing for acids, follow out in general the same scheme as above (page 84).

There are slight deviations only in the tests for phosphates, chlorides and iodides.

After having ascertained that the substance is neither a carbonate, sulphide, sulphate, borate nor nitrate or iodide (comp. page 86), proceed as follows:

To detect Phosphoric Acid, add to a few drops of original solution a drop or two of nitric acid conc. and 4 or 5 vols.

of ammonium molybdate. A yellow precipitate, or at least a yellow coloration of the fluid after a *gentle* heating, indicates (comp. **XX.**, 3) a.....**Phosphate.**

The above method in the hands of beginners being liable to give erroneous results, additional evidence should be produced by way of precipitating with ammonio-magnesium phosphate. This test cannot be made in the usual way (page 86), as it requires a strongly ammoniacal solution. Ammonium hydrate, instead of decomposing the phosphate (with the formation of hydroxide of the base and ammonium phosphate), simply re-precipitates the original substance from its solution in free acid, leaving behind a barren liquid, *i. e.*, the metal of the substance interferes with the ordinary way of testing, and must first be removed.

1. If the the metal found is precipitated by hydrogen sulphide from acid solution, or if it is iron, nickel, cobalt, manganese or zinc, remove the metal by respectively hydrogen or ammonium sulphide and filter off the metallic sulphide. The filtrate contains ammonium phosphate, and may be tested according to page 86 for a.....**Phosphate.**

2. If the metal found is aluminium or chromium, ammonium sulphide would throw down the phosphate. No such precipitate will form if tartaric acid and then excess of ammonium hydrate is added to original solution. Test this clear solution according to page 86 for a....**Phosphate.**

3. If the metal found is barium, strontium or calcium, remove it by dil. sulphuric acid and alcohol. Test the filtrate with excess of ammonium hydrate and magnesium sulphate for a.....**Phosphate.**

4. If the metal is magnesium, moisten some of the original *substance* with a com. sol. of silver nitrate. A yellow coloration produced indicates a.....**Phosphate.**

To detect a **Chloride or Iodide**, it is necessary to dissolve

the substance in nitric acid: Silver nitrate then will produce a curdy precipitate which, after filtering and washing, is to be washed through the perforated filter into a test tube, placed under the funnel.

a. Precipitate dissolves in ammonium hydrate on gentle warming. Substance is a.....**Chloride.**

b. Precipitate does not dissolve. Substance is an .....**Iodide.**

In dissolving a substance in hydrochloric acid observe whether chlorine is liberated on boiling. In this case the substance is either a peroxide or chromate (comp. **VI.**, **XVIII.**, **XXII.**).

SECTION No. 3.

SIMPLE SUBSTANCES, INSOLUBLE (OR NEARLY SO) IN BOTH WATER AND ACIDS.

The sulphates of calcium, strontium, barium and lead, chloride and iodide of silver, ignited chromic acid, and also charcoal and sulphur belong to this class; the latter two may be easily recognized from their color and their behavior before the blowpipe.

Pour some ammonium sulphide on the substance:

1. *It remains unchanged;* no lead or silver compound.

a. Original substance is green, and gives a yellow fused mass with sod. carb. and nitre before blowpipe on platinum foil .....**Chromic Oxide.**

b. Original substance is white. Boil a small knife-point full of the *fine* powder with a conc. sol. of sodium carbonate for about 10 minutes; pour off the supernatant liquid and boil once again with more of the sodium carbonate solution; filter, wash the residue on the filter well and dis-

solve it, by pouring on dil. hydrochloric acid. Test this filtrate for barium, strontium, calcium (page 84). The sodium carb. sol. poured off the first time, to be tested for sulphuric acid (page 86).

2. *Ammonium sulphide turns the substance black;* fuse the substance with sod. carb. on charcoal before the blowpipe. The result is:

a. A malleable metallic globule and a fusion which blackens moist silver. Substance is.....**Lead Sulphate.**

b. A *metallic globule* and a fusion which does *not blacken moist silver.* In this case fuse some of the substance with sod. carb. on porcelain cover, dissolve the fusion in water and filter. Dissolve the well washed residue in nitric acid and test with hydrochloric acid for SILVER. Acidulate filtrate obtained above with nitric acid and test one part with silver nitrate for CHLORINE, the other one with starch paste and dil. fuming nitric acid for IODINE (page 86).

CHAPTER II.

EXAMINATION OF COMPLEX SUBSTANCES SOLUBLE IN WATER OR IN ACIDS.

It is to be understood that of the elements treated on hitherto, chromium and strontium are excluded, and that of the metal tin, antimony and arsenic, only one at a time shall be present; as also of the metals nickel and cobalt.

A. Bases.

OUTLINE OF THE PROCESS.

Heat a small knife-point full of the substance in a test tube one-third full of water. If it dissolves completely add some 6-10 drops of hydrochloric or nitric acid. If the substance is wholly or partly insoluble heat a small knife-point full with hydrochloric or nitric acid in the manner indicated on page 84. (Sec. 2.)

To one-third of this solution add a little

Hydrogen Sulphide.

If a precipitate is formed add hydrogen sulphide *until the solution* AFTER SHAKING *smells of it distinctly,* warm and filter. The precipitate may contain TIN, ARSENIC, ANTIMONY, MERCURY, LEAD, SILVER, COPPER, BISMUTH and CADMIUM. The filtrate may contain the other heavy metals, metals of the earths, alkaline earths and alkalies.

Wash the precipitate on the filter by pouring upon it repeatedly hot water and examine it according to.....I.

Add a little more hydrogen sulphide to the filtrate in order to ascertain whether or not everything precipitated

has been removed; then add some ammonium chloride, supersaturate with ammonium hydrate and, no matter whether this produces a precipitate or not, add:

Ammonium Sulphide,

Drop by drop as long as a reaction can be observed. If hydrogen sulphide failed to produce a precipitate, reject the (thus diluted) liquid and add to a second third of the original solution an excess of ammonium hydrate and then ammonium sulphide.

The precipitate obtained in either way may contain NICKEL, COBALT, IRON, MANGANESE, ZINC and ALUMINIUM, and if PHOSPHORIC or BORIC acid are present, also MAGNESIUM, CALCIUM and BARIUM. Warm this precipitate with the liquid, filter, wash *well* and examine the precipitate according to.....**II.**

To the filtrate from the ammonio-sulphide precipitate, or if no such precipitate was formed, to the original liquid add:

Ammonium Carbonate.

A precipitate obtained will contain CALCIUM and BARIUM. Filter off and examine precipitate according to.....**III.**

Examine the filtrate from this precipitate, or if none was formed, the clear liquid itself with ammonium hydrate and:

Sodium Hydrogen Phosphate.

A precipitate indicates **Magnesium**; see.....**IV.**

Examination for **Alkali Metals** according to..**V.** and **VI.**

I. The precipitate obtained by Hydrogen Sulphide may be:

1. **White** from separated sulphur, which is an indication of a ferric salt (comp. page 14), or:

2. **Yellow, Brown or Black.** Filter off precipitate and

wash it *well;* perforate the bottom of the filter with a match or a glass rod, and rinse the precipitate into a test tube (placed under the funnel) by the aid of a jet of water from the wash bottle. †Allow precipitate to settle, pour off the supernatant water, and *warm* precipitate with *yellow* ammonium sulphide. This will dissolve the precipitate either wholly or partially or not at all.

If the precipitate dissolves perfectly, only one of the metals, *tin, antimony* or arsenic, can be present and the examination is conducted according to.....**A.**

If the precipitate does not dissolve, or only partially, filter off the undissolved portion and

Examine filtrate, according to.....**A.**

Examine residue, according to.....**B.**

A. Tin, Antimony or Arsenic.

Dilute the filtrate or the ammonium sulphide solution well with water and acidulate with hydrochloric acid. The precipitate obtained is:

a. *White* from separated sulphur, merely the result of decomposition of ammonium sulphide.

b. *Yellow* or reddish, and may contain *tin, antimony* or *arsenic.* Filter off precipitate, wash well, and rinse one-half of it from perforated filter into a test tube. Allow precipitate to settle, pour off supernatant water as completely as possible, and dissolve precipitate by boiling with a very *small* quantity of aqua regia. Divide solution into three parts. *To one part add* 3 vols. of conc. oxalic acid and hydrogen sulphide.

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†Note.—Sometimes the hydrogen sulphide precipitate, after having been rinsed from perforated filter, will settle very slowly. Addition of a few drops of hydrochloric acid and gentle heating will shorten very much the time required for settling.

1a. Nothing is precipitated.....**Tin.**

2a. A precipitate forms again: The metal is *arsenic* or *antimony*. Add hydrogen sulphide to a *second part of the aqua regia solution.* The precipitate formed redissolves on warming with ammonium carbonate. Test some of the precipitate left on perforated filter before the blowpipe on charcoal. A garlic-like smell is given off.....**Arsenic.**

3a. The third portion of the aqua regia solution becomes turbid on the addition of a large quantity of water. The precipitate on perforated filter gives a strong white coating on charcoal before the blowpipe.....**Antimony.**

B. Mercury, Lead, Silver, Copper, Bismuth and Cadmium.

The sulphides of these metals constitute that portion of *the hydrogen sulphide precipitate which is insoluble in ammonium sulphide.* Wash this residue well and rinse it from perforated filter into a test tube; allow to settle, pour off the supernatant water and boil with *a little* conc. nitric acid. The sulphides will either dissolve completely (leaving only a dirty yellow mass of sulphur floating in the nitrate solution), or a black or gray powder will remain, which sinks to the bottom rapidly.

a. Filter off this *insoluble residue;* wash, rinse from perforated filter, etc., and dissolve in the least possible amount of aqua regia. Immerse a copper turning into this solution, after previous dilution with water. The copper will turn white from.....**Mercury.**

b. *The filtrate* (**a**) from the separated sulphur or black insoluble residue, may give a white precipitate with dil. sulphuric acid and a little alcohol.....**Lead.**

c. The filtrate from **b**, or, if sulphuric acid and alcohol did not precipitate anything, the liquid itself gives a white curdy precipitate with hydrochloric acid.....**Silver.**

d. Supersaturate the filtrate from **c**, or, if hydrochloric acid produced no precipitate, the liquid itself with the least possible amount of ammonium hydrate. The result is:

1a. A blue solution.....**Copper.**

2a. (With or without blue solution) a white precipitate. Filter and wash the same and dissolve it on the filter with a few drops of hot hydrochloric acid. This solution will give a white precipitate when largely diluted with water .....**Bismuth.**

e. †The ammoniacal solution (**d** 1a) may contain cadmium as well as copper. In this case the addition of sodium hydrate, with heating, will produce a white precipitate, which after filtering and washing is dissolved by hydrochloric acid. Hydrogen sulphide precipitates from this solution yellow sulphide of.....**Cadmium.**

II. ‡***The Ammonium Sulphide precipitate*** is filtered off and washed well, keeping it covered with hot water all the time. Dissolve precipitate on the filter with the *least possible* amount of hot dil. hydrochloric acid (1 vol. acid+4 vols. water). Pour the filtrate back on the filter repeatedly, after having heated it each time to boiling. This will give a

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†Note.—If solution **d** contains a large amount of free acids, the ammoniacal copper-cadmium sol. may be surcharged with ammonium salts to such an extent as to prevent sodium hydrate from producing any precipitate. In this case precipitate the cadmium (with or without copper) by hydrogen sulphide, and test precipitate before blowpipe on charcoal for cadmium. If no copper is present cadmium may be detected by hydrogen sulphide in the ammoniacal filtrate from the bismuth precipitate.

‡Note.—If the substance under examination contains a large amount of metals of the hydrogen sulphide group, the filtrate from their sulphides will be a very dilute solution of the following group metals, and should be concentrated in a porcelain dish before addition of ammonium hydrate and ammonium sulphide.

clear filtrate, free from hydrogen sulphide (which if not removed would eventually interfere with the following tests):

1. †**If a black residue is left on the filter** either cobalt or nickel is present; after washing out, test as follows:

a. A small quantity of it colors the borax bead blue ..... **Cobalt.**

b. Rinse precipitate from perforated filter, etc., and dissolve in the least possible amount of nitric acid. Sodic carbonate produces a light green precipitate in this solution ..... **Nickel.**

2. **The Filtered HCl Solution of the Ammonium Sulphide Precipitate,** if the latter was black, must be boiled with several drops of nitric acid in order to convert any iron that may be present into a ferric salt. Hence, omit the addition of nitric acid if precipitate was not black. This solution *may contain iron, manganese, aluminium and zinc,* and, if substance contains phosphoric or boric acid, also *barium, calcium and magnesium.* The method of examination being comparatively complicated and tedious in the latter case, but simple in the absence of phosphoric (and boric) acid, their presence or absence and the course to pursue should be determined at this moment. To detect phosphoric acid in solution 2, dilute a few drops of it with water, add a drop or two of nitric acid and 10-15 drops of ammonium molybdate and warm (comp. **XX.**, 3). (‡Test for boric acid according to page 116.)

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†Note.—The only safe method to determine both cobalt and nickel in one substance is as follows: Dissolve the black precipitate in nitric acid, slightly supersaturate the liquid with potassium hydrate, add a sol. of potassium-nitrite and acidulate with acetic acid. Filter off the precipitate of potassium cobaltic nitrite, which will form after a time and throw down the nickel in filtrate with sodium carbonate.

‡Note.—The original in this paragraph, does not mention boric acid, apparently assuming that, as a rule, the

A. If no Phosphoric (or Boric) Acid is present, add an excess of sodium hydrate: a permanent precipitate *may contain manganese and iron,* the alkaline liquid *aluminium and zinc.*

a. Filter off and wash the precipitate; ignite a little of it with sodic carbonate and nitre on platinum foil. The fused mass is green.....**Manganese.**

b. Dissolve the greater part in a *small* amount of hydrochloric acid. Potassium ferrocyanide gives in this solution a dark blue precipitate.....**Iron.**

c. †To part of the alkaline filtrate add ammonium chloride. A precipitate indicates.....**Aluminium.**

d. Add some hydric sulphide to the balance. A precipitate shows .....**Zinc.**

B. If Phosphoric (or Boric) Acid have been found, the *solution may contain barium, calcium and magnesium* besides the metals given under **A.** First, barium and calcium are removed as sulphates.

a. Add to solution 2 (page 108) dilute sulphuric acid. Precipitate indicates .....**Barium.**

b. Filter off such precipitate and add an equal vol. of alcohol to the filtrate. Precipitate (perhaps only on shaking) .....**Calcium.**

c. Reject wash water from **b**; heat (not boil) the filtrate

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salts are so selected that the ammonium sulphide precipitate does not contain boric acid: to always conduct examination according to B. might confuse beginners and a previous accurate test for Boron be too difficult for them.

†Note.—If the sodium hydrate solution was not quite clear, filter the alkaline aluminium-zinc solution before adding hydrogen sulphide or ammonium chloride. Warming the liquid moderately for a time, after the addition of these reagents causes the transparent precipitate to shrink together and it is consequently more easily seen.

from **b** for quite a time with excess of sodium hydrate and filter off any permanent precipitate: *ferric, manganous,* and *magnesium hydrates* (**d**).

1a. The alkaline filtrate, containing the **Aluminium and Zinc** (as well as the phosphoric and boric acid of the solution) is to be tested for the two metals as above under **A.**

D. The precipitate (**d**) may contain iron, manganese and magnesium.

1a. †Test a small portion of it, by fusing with sod. carb. and nitre, green fusion.....**Manganese.**

2a. The greater portion of the precipitate should be washed well and then dissolved on the filter with a little dil. hydrochloric acid. Divide this solution into two parts:

Test one part with potassic ferrocyanide. A blue precipitate indicates .....**Iron.**

3a. From the balance of solution precipitate iron and manganese with ammonium hydrate and sulphide; filter off and precipitate from filtrate with sodium hydrogen phosphate .....**Magnesium.**

Unless the barium, calcium and magnesium were carried into the ammonium sulphide precipitate by phosphoric (or boric) acid, they will be found in the remaining two subdivisions.

III. The Ammonium Carbonate precipitate may contain BARIUM and CALCIUM. Wash it on the filter and dissolve by addition warm dil. hydrochloric acid. Add dil. sulphuric acid to the solution:

a. A precipitate forms at once.....**Barium.**

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†Note.—Chromium, in absence of manganese, may be found best in salt mixtures by fusing some of the ammonium sulphide precipitate with sod. carb. and nitre on platinum foil. If chromium is present, the fusion is yellow and the solution in water is of the same color.

b. Filter off that precipitate and add to the filtrate ammonium hydrate and oxalic acid. A precipitate indicates ..... **Calcium.**

IV. ***†The precipitate obtained by Sodium Hydrogen Phosphate indicates*** ..... **Magnesium.**

V. ***To test for Ammonium,*** boil the original substance with sodium hydrate. Any ammonia escaping is easily recognized by its smell and its turning moist red litmus paper blue ..... **Ammonium.**

VI. ***‡To test for Potassium and Sodium,*** remove in a portion of original solution all constituents thrown down by hydrogen sulphide, ammonium sulphide and ammonium carbonate; evaporate filtrate to dryness in a porcelain dish, and heat residue until all ammonium salts are volatilized. A residue obtained:

a. Colors a Bunsen and spirit lamp flame yellow.. ..... **Sodium.**

b. Colors a flame violet ..... **Potassium.**

B. Acids.

The examination of above substances for acids is conducted in the same manner as given in the analysis of *"simple substances"* (comp. pages 84 and 92). *Special attention must be paid to the rules laid down on page 68.*

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†Note.—Before precipitating magnesium, prove with oxalic acid that barium and calcium have been thrown down completely.

‡Note.—If potassium and sodium are present at the same time, the yellow sodium flame will completely hide the violet potassium color. The latter may be detected through a blue glass (cobalt glass). Potassium may also be detected as potassium bitartrate: Dissolve the residue from evaporation in a few drops of warm water and filter. Add to filtrate a drop or two of sodium carbonate and then tartaric acid sol. A precipitate (perhaps only upon shaking) indicates ..... Potassium.

1. **Carbonates** and **Sulphides** are recognized upon heating with hydrochloric acid (page 84) odor or effervescence.

2. Heating the substance with conc. sulphuric acid and a copper turning (brown fumes), or with hydrochloric acid and sol. indigo (fading of color), indicates the presence of a ..... **Nitrate.**

3. †Add **barium chloride** to the highly diluted solution which has been acidulated with hydrochloric acid. A white precipitate shows the presence of a..... **Sulphate.**

4. To detect boric acid, remove with hydrogen sulphide and with sulphuric acid and alcohol all these constituents which color the flame (copper, barium and calcium) and evaporate the filtrate to dryness. Moisten the residue with conc. sulphuric acid and introduce the pasty mass on platinum wire into the edge of a flame. A green color indicates a ..... **Borate.**

5. To prove the presence of **chlorides** and **iodides** dissolve the substance in nitric acid, add silver nitrate to the diluted solution and filter off. Rinse the well-washed precipitate from the filter and warm slightly with ammonium hydrate. The precipitate dissolves:

a. Completely—presence of only a..... **Chloride.**

b. Imperfectly. Filter off the residue, and acidulate filtrate with nitric acid. A precipitate indicates a.. **Chloride.**

6. To prove the presence of iodine, add to the well diluted original solution a little starch paste and a drop or two of dil. nitric acid fuming (comp. **XXIV.**, 4, page 66). A blue color proves the presence of an..... **Iodide.**

7. To a drop of original solution, diluted with a little water, add a few drops of nitric acid and excess of ammonium molybdate and warm gently. A yellow precipitate (or

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†Note.—If hydrochloric acid produces a precipitate, filter off before adding barium chloride.

at least coloration) shows the presence of a.....**Phosphate.**

Or: Remove with hydrogen sulphide, sulphuric acid and alcohol all the elements precipitated in this way. Concentrate the filtrate if necessary, and heat with excess of conc. *sodium hydrate* and filter off precipitate. The phosphoric acid is concentrated in this alkaline liquid (comp. pages 92, 94). As it also contains the aluminium of the substance it is necessary to acidulate the liquid with tartaric acid before it may be tested with ammonium hydrate and magnesium sulphate a precipitate.....**Phosphate.**

C. Alloys.

In analyzing alloys it is useless, of course, to test for acids as well as in the majority of cases for the metals of the alkalies and alkaline earths, as they very rarely occur in alloys.

Use conc. nitric acid to dissolve alloys, warming a few pieces (as small as possible) of the alloy until the metal has disappeared.

If there is a white residue, it is either stannic or antimonous acid, which should be filtered off, washed well and dissolved by boiling with hydrochloric acid. Examine the solution obtained according to page 102.

The solution of the alloy in nitric acid contains all other metals; dilute somewhat with water and precipitate the *silver,* that may be present, by addition of a few drops of hydrochloric acid. Filter off the curdy precipitate and throw down the *lead* present as sulphate by the addition of dil. sulphuric acid and some alcohol. The filtrate of this precipitate is examined with hydrogen and ammonium sulphide in the ordinary manner described above (page 98).

NAME	DATE DUE
A. E. Shuky	Sept 5, 1916